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DICTIONARY OF ORGANIC COMPOUNDS

VOLUME 4

DICTIONARY OF ORGANIC COMPOUNDS

The Constitution and Physical and Chemical Properties of the Principal Carbon Compounds and their Derivatives, together with the relevant Literature References

VOLUME FOUR

OBACULACTONE — ZYMOSTEROL

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Reference

INTRODUCTION

All the compounds in this dictionary are arranged in strictly alphabetical order. The dictionary is therefore its own index, and any required data and information should be readily accessible. A difficulty sometimes arises, however, on account of the fact that most organic compounds possess more than one name, and questions of orientation introduce a further complication. An alphabetical classification is thus inadequate *per se*, and some further rules are necessary if that otherwise simple system is to possess its maximum utility. In order to facilitate the use of the dictionary an explanation of the general rules which have been adopted and followed throughout the work is given in the following notes.

Arrangement of the Text

Principal References

The majority of compounds can be named in a variety of ways, some popular (or trivial) and others systematic or strictly chemical. But in practice it is found that for any one compound preference is usually given to one particular name, and it is this name which has been selected for the principal reference. It sometimes happens, however, that such a compound is one of a number of isomers having a systematic or chemical name. In certain cases of this kind the general rule has been departed from, the whole series of isomers being grouped together under the systematic name. For example, Orthanilic, Metanilic, and Sulphanilic Acids are not separated under letters O, M, and S, but are given together under letter A as Aniline-*o*-, *m*-, and *p*-sulphonic Acids respectively. Similarly Gamma Acid, J-Acid, M-Acid, and S-Acid are included with the other Aminonaphtholsulphonic Acids. Trade and proprietary names are not used for the principal references, except where they come within the general rule as being in common use or where, usually for reasons of complex structure, no other names are feasible.

The name selected for a parent compound is, with a few exceptions, retained for all its derivatives. For example, Alizarin having been chosen for 1 : 2-Dihydroxyanthraquinone, all the derivatives of the latter will be found as derivatives of Alizarin.

Cross-References

A generous number of cross-references, sometimes as many as three or four, are given, by means of which the principal references can readily be found. For instance *N*-Carboxyurea, Carbamylcarbamic Acid, and Ureidoformic Acid are given as cross-references to Allophanic Acid. Where cross-references are given for a parent compound, these are not, in general, repeated for the derivatives. Thus Diphenyl Ketone will be found as a cross-reference to Benzophenone, but Amino- and Bromo-diphenyl Ketones are not given as cross-references to Amino- and Bromo-benzophenones. Owing to their large number many nitriles are not included under Cyano– : those not so given will be found either under their own name (e.g. Acetonitrile, Benzonitrile) or under the parent acid.

Formulæ

The full constitutional formula is given for each compound with, of course, the exception of those whose constitutions have not yet been ascertained. In the case of many compounds of the carbohydrate, steroid, and polyterpene classes, the constitution is represented by simple skeletal structures and configuration by heavy or dotted lines in conformity with modern practice. Where there is a group of isomers, however, it is considered to be sufficient in most cases to set out the full formula for the first member only. This applies also to the molecular formula and molecular weight. For instance, the complete formula is given for *o*-Chlorotoluene, but none for the *m*-, and *p*-, which follow. For acyclic compounds, however, particularly those with long or branched chains or with varying degrees of unsaturation, orientation is less simple, and full formulæ are given in every case, *e.g.*, the Hexenols.

The order of the elements in the molecular formulæ is C H O N Cl Br F S P, other elements following in alphabetical order. In calculating molecular (formula) weight the following approximate atomic weights have been employed : C 12, H 1, O 16, N 14, Cl 35·5, Br 80, I 127, F 19, S 32, P 31, Fe 56, Mg 24, Pb 207, Se 79, Si 28, and for other elements the whole number nearest to the atomic weight adopted by the International Committee on Atomic Weights in 1942.

Derivatives

Functional derivatives, as distinct from substitution compounds, are given under the parent compound except where they are sufficiently important to justify separate inclusion as principal references. Thus under an acid will be found its esters, halides, anhydride, amide, nitrile, anilide, etc. Esters in which the alcohol or phenol component is of more importance than the acid component are given under the alcohol or phenol, as, *e.g.*, in the case of the acetates and benzoates of Borneol, Resorcinol and *sec.*-*n*-Octyl alcohol. Under a base are given its salts, acyl derivatives, addition compounds, etc.; under an alcohol or phenol, the ethers and certain esters (*v. supra*); under an aldehyde or ketone, the acetals, oximes, semicarbazones, phenylhydrazones, etc.

Literature References

The literature references relate primarily to the best method of preparation, but in some cases publications are cited which refer to earlier methods of preparation or which contain useful summaries or bibliographies. Where considerations of molecular structure or constitution are of importance, reference is made to the most recent views on the subject. This is particularly the case with many of the complex naturally occurring compounds. To some of the very common compounds, *e.g.*, Acetone, Acetic acid and Ethyl alcohol, no useful references can be given.

The journal abbreviations used are listed in the Table on p. xiii. They are largely those adopted for the *Chemical Abstracts* of the American Chemical Society (Index to *Chemical Abstracts*, **45**, 1951).

Nomenclature and Orientation

In the introduction to Richter's Lexicon (1910) the following passage occurs: " Of the many difficulties which, in writing this work, presented themselves, the greatest was undoubtedly the question 'which nomenclature is to be adopted for the sake of uniformity and clearness.' " The vast number of compounds already known when that was written has since been continuously added to, and new types of compound have appeared, thus rendering the problem of nomenclature more difficult and complex to-day than it was in 1910. The only system which can be regarded as having the merit of completeness is that of Beilstein, but it is very complicated, and possesses other features which reduce its value as a practical scheme. The Geneva system, although occasionally utilised for aliphatic compounds, has never found universal favour. Many attempts have been made in recent years to introduce new systems both of nomenclature and of orientation, but none of these has been exclusively used in this Dictionary. Preference has usually been given to the nomenclature and orientation employed in the original publication, but, in cases where ambiguity may arise, adjustment has sometimes been necessary in the interests of clarity.

In order to avoid re-setting in a large number of compounds scattered over the 3,000 pages of this edition, the recently introduced convention of using small roman capitals, D, L, in place of italic, *d*-, *l*-, for optically active compounds, has not been adopted except in a few instances.

Nomenclature

The prefixes cyclo-, homo-, iso-, etc., are treated as part of the name. Thus Cyclopentane, Homocatechol, Isophthalic Acid, are to be found under letters C, H, I, respectively. The prefixes allo-, epi-, and nor-, have also in general been prefered as part of the name, but where this has not been convenient sufficient cross-references have been given. An exception is made in the case of pseudo-, which is represented by the Greek letter ψ, ψ-Aconitine (Pseudo-aconitine), for instance, being included under letter A and not letter P. The prefixes benz- and naphtha- are used for condensed (fused) benzene and naphthalene rings, not benzo- and naphtho-. *E.g.*, Benzthiazole, Benzcarbazole, Naphthafluorene. For compounds containing the fused naphthalene ring and for whose names both benz- and naphtha- have been used, the preference has been given to the former in principal references, *e.g.*, Benzcoumarin not Naphthacoumarin, Benzacridine not Naphthacridine. For acetylenic compounds -in- has been replaced by -yn-, *e.g.*, Butyne not Butine, Hexynol not Hexinol, and similarly ethynyl instead of ethinyl.

All " double " derivatives take the prefix di-. *E.g.*, Diallyl sulphide, not Allyl sulphide; Diethyl Ether, not Ethyl Ether; Dimethyl sulphate, not Methyl sulphate. The Greek di- is preferred to the Latin bi- or bis-, *e.g.*, di-semicarbazone, di-phenylhydrazone.

In substituted or derived compounds the order in which the substituent group names appear in the compound name is that given in the Table on p. xv, List of Substituents. Thus, for the common substituents the order is, Halogen (F, Cl, Br, I), Nitro, Hydroxy, Amino, Alkyl, Aryl, Acyl. Wherever possible, the aldehyde, carboxyl, and sulphonic groups are preferred as suffixes. *E.g.*, Anthraquinonesulphonic Acid, not Sulphoanthraquinone (but cf. *e.g.*, Disulphobenzoic Acid, Aldehydosalicylic Acid); Chloroanthraquinonecarboxylic Acid, not Chlorocarboxyanthraquinone.

ORIENTATION

Acyclic Compounds. The numbering system is used in preference to Greek letters. The CHO, CN, and COOH groups are generally regarded as substituents and, as such, their carbon atoms are not numbered.

In the *saturated branched chains* numbering commences in the longest chain at the terminal carbon nearest to the side-chain. If there are two or more side-chains or groups of different lengths, numbering commences at the end carbon nearest to the shortest of such side-chains. *E.g.*,

$$\underset{4}{CH_3}\cdot\underset{3}{CH_2}\cdot\underset{2}{\overset{\overset{3'}{CH_3}}{\overset{|}{CH}}}\cdot\underset{1}{CH_3}$$

Isopentane

$$\underset{7}{CH_3}\cdot\underset{6}{CO}\cdot\underset{5}{CO}\cdot\underset{4}{CH_2}\cdot\underset{3}{CH_2}\cdot\underset{2}{\overset{\overset{3'}{CH_3}}{\overset{|}{CH}}}\cdot\underset{1}{CH_3}$$

Acetylisocaproyl

There are two exceptions to the above rule. (1) Where the chain contains a CHO, CN, or COOH group, numbering begins at the carbon carrying this group. *E.g.*,

$$\underset{4}{CH_3}\cdot\underset{3}{\overset{\overset{4'}{CH_3}}{\overset{|}{CH}}}\cdot\underset{2}{CH_2}\cdot\underset{1}{CH_2}\cdot COOH$$

Isocaproic Acid

(2) In the primary alcohols numbering commences at the carbon of the —CH_2OH group. *E.g.*,

$$\underset{4}{CH_3}\cdot\underset{3}{\overset{\overset{4'}{CH_3}}{\overset{|}{CH}}}\cdot\underset{2}{CH_2}\cdot\underset{1}{CH_2}OH$$

Isoamyl Alcohol

In the *unsaturated* compounds the double or triple bonds are given the lowest numbers possible whether the chain is branched or not. Where both double and triple bonds are present, the latter takes the lowest number. *E.g.*,

$$\underset{4}{CH_3}\cdot\underset{3}{CH_2}\cdot\underset{2}{CH}:\underset{1}{CH_2}$$

1-Butylene

$$\underset{4}{CH_3}\cdot\underset{3}{\overset{\overset{4'}{CH_3}}{\overset{|}{CH}}}\cdot\underset{2}{CH}:\underset{1}{CH_2}$$

3-Methyl-1-butylene

$$\underset{4}{CH_2}:\underset{3}{CH}\cdot\underset{2}{C}\vdots\underset{1}{CH}$$

Vinylacetylene

The rule applies to compounds containing the CHO, CN, or COOH groups provided the name of the parent hydrocarbon is retained. *E.g.*,

$$HOOC\cdot\underset{4}{CH_2}\cdot\underset{3}{CH_2}\cdot\underset{2}{\overset{\overset{COOH}{|}}{C}}:\underset{1}{CH_2}$$

1-Butylene-2 : 4-dicarboxylic Acid
(not 3-Butylene-1 : 3-dicarboxylic Acid)

but in all other cases the rule for saturated compounds is followed, viz. numbering begins at the carbon carrying the CHO, CN, or COOH group or, in the case of primary alcohols, at the carbon of the CH_2OH group. Thus

$$\underset{3}{CH_2}:\underset{2}{CH}\cdot\underset{1}{CH_2}\cdot COOH$$

Vinylacetic Acid

$$\underset{17}{CH_3}\cdot[CH_2]_7\cdot\underset{9}{CH}:\underset{8}{CH}\cdot[CH_2]_6\cdot\underset{1}{CH_2}\cdot COOH$$

Oleic Acid

$$\underset{3}{CH_2}:\underset{2}{CH}\cdot\underset{1}{CH_2}OH$$

Allyl Alcohol

$$\underset{7}{CH_3}\cdot\underset{6}{\overset{\overset{CH_3}{|}}{C}}:\underset{5}{CH}\cdot CH_2\cdot\underset{3}{CH_2}\cdot\underset{2}{\overset{\overset{CH_3}{|}}{CH}}\cdot\underset{1}{CH_2}\cdot CHO$$

Citronellal

In the case of carbon chains interrupted by O (ethers), NH (secondary amines), S (sulphides), etc., numbering commences at the carbon atoms attached to the oxygen, nitrogen, sulphur, etc. *E.g.*,

$\underset{4'}{CH_3}\cdot\underset{3'}{CH_2}\cdot\underset{2'}{CH_2}\cdot\underset{1'}{CH_2}\cdot NH\cdot\underset{1}{CH_2}\cdot\underset{2}{CH_2}\cdot\underset{3}{CH_2}\cdot\underset{4}{CH_3}$
Dibutylamine

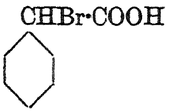
$Cl\underset{2'}{CH_2}\cdot\underset{1'}{CH_2}\cdot S\cdot\underset{1}{CH_2}\cdot\underset{2}{CH_2}Cl$
2 : 2′-Dichlorodiethyl sulphide

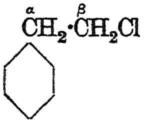
Homocyclic and Heterocyclic Compounds. As stated above, no one system of orientation of ring compounds has been exclusively followed, and no new departures have been made from those already in use. Where there is any doubt numbers have been inserted in the constitutional formulæ of the parent compounds. The numbering adopted for any particular ring system can therefore be readily ascertained by reference to the text. In the methylbenzenes (*i.e.* toluene and its derivatives) numbering begins in most cases at the carbon to which the methyl group is attached.

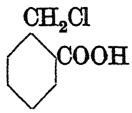
In heterocyclic rings containing both oxygen (or sulphur) and nitrogen the lowest number is given to the oxygen (or sulphur). *E.g.*, in the oxazoles (or thiazoles) numbering commences at the oxygen (or sulphur).

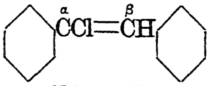
Where there is a choice of numbering of polysubstituted compounds the lowest number is, wherever possible, assigned to the functional groups (OH, NH_2, etc.). *E.g.*, 5-Chloro-1-aminoanthraquinone, not 1-Chloro-5-aminoanthraquinone, such a compound being regarded as a chloro derivative of aminoanthraquinone rather than an amino derivative of chloroanthraquinone. Similarly 6-Bromo-3-hydroxybenzyl Alcohol, not 2-Bromo-5-hydroxybenzyl Alcohol.

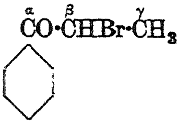
Aromatic-aliphatic (*aralkyl*) *compounds.* In the aralkyl compounds the general rule given above for the numbering of aliphatic chains has been departed from, the carbons in the aliphatic part of the molecule being designated by Greek letters in order to avoid confusion with the numbers of the rings. *E.g.*,

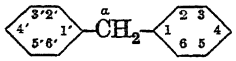

α-Bromophenylacetic Acid

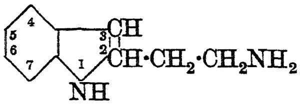

β-Chloroethylbenzene


ω-Chloro-*o*-toluic Acid

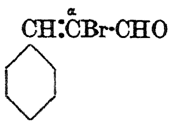

α-Chlorostilbene

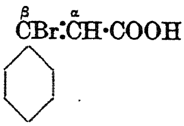

β-Bromopropiophenone

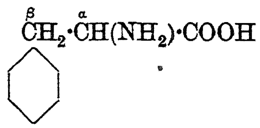

Diphenylmethane


2-[ω-Aminoethyl]-indole

The letters begin at the carbon attached to the ring except in the case of the aromatic-aliphatic acids, aldehydes, nitriles, or primary alcohols. In these cases lettering begins at the carbon carrying the COOH, CHO, or CN group or at the carbon of the CH_2OH group. *E.g.*,


α-Bromocinnamaldehyde


β-Bromocinnamic Acid


α-Aminohydrocinnamic Acid

In many aralkyl compounds, however, the aromatic radical can, from the point of view of the name, be regarded merely as a substituent. In such cases the numbering of the parent aliphatic compound is retained provided no ambiguity is likely to result. Thus,

$C_6H_5\cdot CH{:}CH\cdot CH_2\cdot CH_2\cdot COOH$
3- (not γ-) Benzylidenebutyric Acid

$CH_3\cdot CH_2\cdot CH(C_6H_5)\cdot CH_2C_6H_5$
1 : 2-(not αβ-) Diphenylbutane

$(C_6H_5)_2CH\cdot CH_2\cdot COOH$
2 : 2-(not ββ-)Diphenylpropionic Acid

General Remarks

In the descriptive matter in the text the same order has been followed in all cases, viz.: sources, where of interest; physical properties (melting point, boiling point, solubility, density, refractive index, heat of combustion, optical rotation, etc.) : chemical properties (typical reactions, analytical tests, etc.). The data for derivatives then follow under separate sub-headings.

As regards abbreviations, these have been used freely in the text, but all abbreviations and contractions, such as Me, Et, Ph, CO_2H, etc., have been rigidly excluded from the constitutional formulæ.

As would be expected, the original literature reveals numerous instances of divergent and sometimes contradictory data for the same compound. As far as possible palpable inconsistencies have not been recorded, but where a choice could not be safely made the alternative data, *e.g.*, melting and boiling points, densities, etc., are given in parentheses. In some cases the information given in the literature is so scanty and uncertain that it has been considered advisable to omit the compound altogether. Nevertheless, although in some instances the recorded facts regarding chemical and physical properties are very meagre, the compound has been included by virtue of other valuable information contained in the literature references, such as method of preparation or proof of constitution.

TABLE OF ABBREVIATIONS

A	Acid (A_2, two mols of acid).
Å	Angstrom unit. (10^{-8} cm.).
Abs. EtOH	Absolute alcohol.
AcOH	Acetic acid.
Ac_2O	Acetic anhydride.
AcOEt	Ethyl acetate.
Add.	Additive.
Add. comp.	Addition compound, molecular compound, adduct.
Addn.	Addition.
A.G.F.A.	Aktien-Gesellschaft für Anilinfabrikation.
Alc.	Alcohol, alcoholic.
Alc. NH_3	Alcoholic ammonia.
Al.Hg.	Aluminium amalgam.
Alk.	Alkaline.
[α]	Specific rotation.
Amorph.	Amorphous.
Anhyd.	Anhydrous.
Aq.	Aqueous.
as.	Asymmetric.
Atm.	Atmosphere(s), atmospheric.
B	Base (B_2, two mols of base).
Badische	Badische Anilin und Sodafabrik.
Belg. P.	Belgian Patent.
B.D.C.	British Dyestuffs Corporation.
Bibl.	Bibliography.
B.p.	Boiling point.
B.P.	British Patent.
c.	Concentration.
C_p	Constant pressure.
C_v	Constant volume.
Cal.	Calories.
Can. P.	Canadian Patent.
Col.	Colour, coloration.
Comb.	Combustion.
Comp.	Compound.
Conc.	Concentrated.
Corr.	Corrected.
Crit.	Critical.
Cryst.	Crystals, crystalline, crystallise.
$(COOH)_2$	Oxalic acid.
$(CH_2COOH)_2$	Succinic acid.
D	Density.
D	Dextro-.
d	Dextrorotatory.
dl	Racemic. Optically inactive by external compensation.
Decomp.	Decomposed, decomposition.
Deriv.	Derivative.
Dil.	Dilute, dilution.
Diss.	Dissolves, dissolved.
Dist.	Distil, distillation.
D.R.P.	German Patent.
Et	Ethyl.
Et_2O	Ether (diethyl ether).
EtOH	Ethyl alcohol.
Evac.	Evacuated.
Fluor.	Fluoresces, fluorescence.
F.p.	Freezing point.
F.P.	French Patent.
Form.	Formation.
γ	10^{-6} gm. or 10^{-3} mgm. (microgrammes).
gm.	Gramme(s).
Hyd.	Hydrolyses, hydrolysed, hydrolysis.
i	Optically inactive by internal compensation.
I.C.I.	Imperial Chemical Industries.
I.G.	Interessen Gemeinschaft Farbenindustrie Aktien-Gesellschaft.
Insol.	Insoluble.
I.U.	International Unit.
Jap. P.	Japanese Patent.
k	Dissociation constant.
l	Lævorotatory.
Liq.	Liquid.
m	Meta (position).
Max.	Maximum.
Me	Methyl.
MeOH	Methyl alcohol.
Me_2CO	Acetone.
Min.	Mineral (inorganic).
Misc.	Miscible.
M.L.B.	Meister, Lucius & Brüning.
mm.	Millimetre(s).
Mod.	Moderately.
Mol.	Molecule, molecular, molar.
M.p.	Melting point.
ms	Meso (position).
MW	Molecular weight (formula weight).
mgm.	Milligramme(s).
mμ	Millimicron(s). (10^{-7} cm.).
n	Normal (chain).
n_D	Refractive index (D line, etc.).
NaHg	Sodium amalgam.
NH_3	Ammonia, aqueous ammonia.
$NH_3.AgNO_3$	Ammoniacal silver nitrate.
o	Ortho (position).
Ord.	Ordinary.
Org.	Organic.
Ox.	Oxidise, oxidation.
p	Para (position).
P	Patent.
pH	$-\log_{10}$ hydrogen ion concentration.
pK	$-\log_{10}$ *k* (dissociation constant).
Part.	Partly, partial.
Pet. ether	Petroleum ether.
$PhNO_2$	Nitrobenzene.
PhOH	Phenol.
Ppd.	Precipitated.
Ppt.	Precipitate.
Pptn.	Precipitation.
Prac.	Practically.
Press.	Pressure(s).
ψ	Pseudo.
Py	Pyridine.
r	Racemic.
Red.	Reduce, reduction.
Ref.	Reference.
Russ.P.	Russian Patent.
S.C.I.	Société pour l'industrie chimique à Basle.
Sec.	Secondary.
Sol.	Soluble, solution.
Spar.	Sparingly.
Sp. gr.	Specific gravity.
Sp. heat	Specific heat.
Spp.	Species.
Suppl.	Supplement.
Sym.	Symmetrical.
Temp.	Temperature(s).
Tert.	Tertiary.
Undecomp.	Undecomposed.
Unsym.	Unsymmetrical.
UV.	Ultraviolet.
Vac.	Vacuum.
Vap.	Vaporisation.
Vol.	Volume.

JOURNAL ABBREVIATIONS

Journals not listed here are given their full titles in the text.

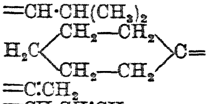
Abbreviation	Full title
Acta Chem. Scand.	Acta Chemica Scandinavica.
Acta Phytochim.	Acta Phytochimica (Japan).
Am. Chem. J.	American Chemical Journal.
Am. J. Pharm.	American Journal of Pharmacy.
Am. J. Sci.	American Journal of Science.
Anales asoc. quím. Argentina	Anales de la Asociación Química Argentina.
Anales soc. españ. fís. quím.	Anales de la sociedad española de física y química.
Angew. Chem.	Angewandte Chemie.
Ann.	Annalen der Chemie.
Ann. chim.	Annales de chimie.
Ann. chim. applicata	Annali di chimica applicata.
Ann. chim. phys.	Annales de chimie et de physique.
Ann. pharm. franç.	Annales Pharmaceutiques Française.
Ann. phys.	Annales de physique.
Ann. Physik	Annalen der Physik.
Ann. Rev. Biochem.	Annual Review of Biochemistry.
Ann. Sci. Univ. Jassy	Annales scientifiques de l'Université de Jassy.
Ann. Trop. Med. and Parasit.	Annals of Tropical Medicine and Parasitology.
Arch. Biochem.	Archives of Biochemistry (name changed to next in 1951).
Arch. Biochem. Biophys.	Archives of Biochemistry and Biophysics.
Arch. Pharm.	Archiv der Pharmazie (und Berichte der deutschen pharmazeutischen Gesellschaft).
Arkiv Kemi, Mineral. Geol.	Arkiv för Kemi, Mineralogi och Geologi.
Atti accad. Lincei	Atti della reale accademia nazionale dei Lincei.
Ber.	Berichte der deutschen chemischen Gesellschaft. Now Chemische Berichte.
Ber. deut. pharm. Ges.	Berichte der deutschen pharmazeutischen Gesellschaft.
Ber. ges. Physiol. exptl. Pharmakol.	Berichte über die gesamte Physiologie und experimentelle Pharmakologie.
Bihang till Svenska Vet.-Akad. Handlingen	Bihang till Kungliga Svenska Vetenskapsakademiens Handlingen.
Biochem. J.	Biochemical Journal.
Biochem. Z.	Biochemische Zeitschrift.
Biol. Zentr.	Biologisches Zentralblatt.
Brit. Chem. Abstracts	British Chemical Abstracts.
Brit. J. Exptl. Path.	British Journal of Experimental Pathology.
Brit. J. Pharmacol.	British Journal of Pharmacology and Chemotherapy.
Bull. acad. sci. U.S.S.R.	Bulletin of the Academy of Sciences of the U.S.S.R.
Bull. Chem. Soc. Japan	Bulletin of the Chemical Society of Japan.
Bull. Imp. Inst.	Bulletin of the Imperial Institute.
Bull. Inst. Phys. Chem. Research, Tokyo	Bulletin of the Institute of Physical and Chemical Research, Toyko.
Bull. sci. acad. roy. Belg.	Bulletin de la classe des sciences, academie royale de Belgique.
Bull. sci. pharmacol.	Bulletin des sciences pharmacologiques.
Bull. soc. chim.	Bulletin de la société chimique de France.
Bull. soc. chim. Belg.	Bulletin de la société chimique de Belgique. Now Bulletin des sociétés chimiques Belges.
Bull. soc. chim. biol.	Bulletin de la société de chimie biologique.
Can. Chem. Met.	Canadian Chemistry and Metallurgy.
Can. J. Research	Canadian Journal of Research.
Chem. Abstracts	Chemical Abstracts (of the American Chemical Society).
Chem. Ind.	Die Chemische Industrie.
Chem. Met. Eng.	Chemical and Metallurgical Engineering.
Chem. News	Chemical News (and Journal of Industrial Science).
Chem. Reviews	Chemical Reviews.
Chem.-Tech. Rundschau	Chemische-Technische Rundschau.
Chem. Trade J.	Chemical Trade Journal (and Chemical Engineer).
Chem. Umschau	Chemische Umschau (auf dem Gebiete der Fette, Oele, Wachse, und Harze). Now Fettchemische Umschau.
Chem. Weekblad	Chemisch Weekblad.
Chem. Zentr.	Chemisches Zentralblatt.
Chem.-Ztg.	Chemiker-Zeitung.
Compt. rend.	Comptes rendus (hebdomadaires des séances de l'académie des sciences).
Compt. rend. acad. sci. U.R.S.S.	Comptes rendus de l'Académie des Sciences de l'U.R.S.S.
Compt. rend. soc. biol.	Comptes rendus des séances de la société de biologie.
Color Trade J.	Color Trade Journal and Textile Chemist.
Dinglers polytech. J.	Dinglers polytechnisches Journal.
Fettchem. Umschau	Fettchemische Umschau.
Gazz. chim. ital.	Gazzetta chimica italiana.
Giorn. chim. applicata	Giornale di chimica applicata.
Giorn. chim. ind.	Giornale di chimica industriale.
Giorn. chim. ind. applicata	Giornale di chimica industriale ed applicata.
Helv. Chim. Acta	Helvetica Chimica Acta.
Ind. Eng. Chem.	IndustrialandEngineeringChemistry.
Jahresber. Fortschr. Chem.	Jahresbericht über die Fortschritte der Chemie.
Japan. J. Chem.	Japanese Journal of Chemistry.
J. Am. Chem. Soc.	Journal of the American Chemical Society.
J. Am. Pharm. Assocn.	Journal of the American Pharmaceutical Association.
J. appl. Chem.	Journal of Applied Chemistry. Formerly Journal of the Society of Chemical Industry.
J. Applied Chem., U.S.S.R.	Journal of Applied Chemistry U.S.S.R.
J. Bact.	Journal of Bacteriology.

J. Biochem. Japan	Journal of Biochemistry of Japan.
J. Biol. Chem.	Journal of Biological Chemistry.
J. Chem. Education	Journal of Chemical Education.
J. Chem. Ind. Japan	Journal of Chemical Industry (Japan). Now J. Soc. Chem. Ind. Japan.
J. Chem. Physics	Journal of Chemical Physics.
J. Chem. Soc.	Journal of the Chemical Society (London).
J. Chem. Soc. Abstracts	Abstracts of the Chemical Society (London).
J. Chem. Soc. Japan	Journal of the Chemical Society of Japan.
J. chim. phys.	Journal de chimie physique.
J. Chinese Chem. Soc.	Journal of the Chinese Chemical Society.
J. Gen. Chem. U.S.S.R.	Journal of General Chemistry, U.S.S.R.
J. gen. Microbiol.	Journal of General Microbiology.
J. Indian Chem. Soc.	Journal of the Indian Chemical Society.
J. Indian Inst. Sci.	Journal of the Indian Institute of Science.
J. Inst. Petroleum	Journal of the Institute of Petroleum.
J. Org. Chem.	Journal of Organic Chemistry.
J. pharm. Belg.	Journal de pharmacie de Belgique.
J. pharm. chim.	Journal de pharmacie et de chimie.
J. Pharm. Soc. Japan	Journal of the Pharmaceutical Society (Japan).
J. Pharmacol.	Journal of Pharmacology and Experimental Therapeutics.
J. Pharm. Pharmacol.	Journal of Pharmacy and Pharmacology.
J. Phys. Chem.	Journal of Physical Chemistry.
J. prakt. Chem.	Journal für praktische Chemie.
J. Proc. Roy. Soc. N.S. Wales	Journal and Proceedings of the Royal Society of New South Wales.
J. Russ. Phys.-Chem. Soc.	Journal of the Russian Physical-Chemical Society.
J. Soc. Chem. Ind. Now J. appl. Chem.	Journal of the Society of Chemical Industry.
J. Soc. Chem. Ind. Japan	Journal of the Society of Chemical Industry (Japan).
J. Soc. Dyers Colourists	Journal of the Society of Dyers and Colourists.
Mem. Coll. Sci., Kyoto Imp. Univ.	Memoirs of the College of Science, Kyoto Imperial University.
Monatsh.	Monatshefte für Chemie und verwandte Teile anderer Wissenschaften.
Naturwiss.	Naturwissenschaften.
Org. Chem. Ind. U.S.S.R.	Promischlennosti Organitscheskoi Chimii, U.S.S.R.
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae.
Pharm. J.	Pharmaceutical Journal and Pharmacist.
Pharm. Weekblad	Pharmaceutisch Weekblad.
Pharm. Ztg.	Die deutsche Pharmazeutische Zeitung.
Pharm. Zentralhalle	Pharmazeutische Zentralhalle.
Phil. Mag.	Philosophical Magazine and Journal of Science.
Physical Rev.	Physical Reviews.
Physiol. Rev.	Physiological Review.
Proc. Acad. Sci., Amsterdam	Proceedings of the Royal Academy of Sciences of Amsterdam.
Proc. Chem. Soc.	Proceedings of the Chemical Society (London).
Proc. Imper. Acad., Tokyo	Proceedings of the Imperial Academy, Tokyo.
Proc. Ind. Acad. Sci.	Proceedings of the Indiana Academy of Science.
Proc. Indian Acad. Sci.	Proceedings of the Indian Academy of Science.
Proc. Roy. Soc.	Proceedings of the Royal Society (London).
Proc. Soc. Exptl. Biol. Med.	Proceedings of the Society for Experimental Biology and Medicine, New York.
Quart. J. Indian Chem. Soc.	Quarterly Journal of the Indian Chemical Society.
Quart. J. Pharm. Pharmacol.	Quarterly Journal of Pharmacy and Pharmacology.
Rec. trav. chim.	Recueil des travaux chimiques des Pays-Bas.
Rev. chim. ind.	Revue de chimie industrielle.
Rev. prod. chim.	Revue des produits chimiques.
Sci. Papers Inst. Phys. Chem. Research, Tokyo	Scientific Papers of the Institute of Physical and Chemical Research (Tokyo).
Sci. Proc. Roy. Dublin Soc.	Scientific Proceedings of the Royal Dublin Society.
Sci. reps. Natl. Tsinghua Univ.	Science Reports of the National Tsinghua University.
Sci. reps. Natl. Univ. Peking	Science Reports of the National University of Peking.
Sitzb. Akad. Wiss. Wien	Sitzungsberichte Akademie der Wissenschaften in Wien.
Svensk Farm. Tids.	Svensk Farmaceutische Tidskrift.
Svensk Kem. Tid.	Svensk Kemisk Tidskrift.
Trans. Faraday Soc.	Transactions of the Faraday Society.
Trans. Roy. Soc.	Transactions of the Royal Society (London).
Trans. Roy. Soc. Canada	Transactions of the Royal Society of Canada.
Z. anal. Chem.	Zeitschrift für analytische Chemie.
Z. angew. Chem.	Zeitschrift für angewandte Chemie. Later Angewandte Chemie, now Die Chemie.
Z. anorg. allgem. Chem.	Zeitschrift für anorganische und allgemeine Chemie.
Z. Chem.	Zeitschrift für Chemie.
Z. Elektrochem.	Zeitschrift für Elektrochemie und angewandte physikalische Chemie.
Z. ges. Naturwiss.	Zeitschrift für die gesamte Naturwissenschaft.
Z. physik. Chem.	Zeitschrift für physikalische Chemie.
Z. physiol. Chem.	Zeitschrift für physiologische Chemie (Hoppe-Seyler).

LIST OF SUBSTITUENTS

In the following table is given a list of the principal substituent groups as they are used in the dictionary.

No.	Group	Name
1	—F	Fluoro
2	—Cl	Chloro
3	—Br	Bromo
4	—I	Iodo
5	—NO	Nitroso
6	—NO_2	Nitro
7	—$N{=}N{\rightarrow}N$	Azido, Triazo
8	—OH	Hydroxy (*followed by* —OCH_3 Methoxy, —OC_2H_5 Ethoxy, —$O\cdot CH_2\cdot O$— methylenedioxy, —OC_6H_5 Phenoxy, —$O\cdot CO\cdot CH_3$, Acetoxy, *etc. in the order of the group attached to the oxygen*)
9	—SH	Mercapto
10	—SO	Thionyl, Sulphenyl
11	—SO_2	Sulphonyl
12	—$SO_2\cdot CH_3$	Mesyl
13	—SCN	Thiocyano
14	=O (in C—CO—C)	Keto
15	>NH	Imino
16	=N·OH	Isonitroso, Oximino
17	—S—	Thio
18	—SO_3H	Sulpho
19	—NH_2	Amino
20	—$NH\cdot C_6H_5$	Anilino, Phenylimino
21	—$NH\cdot C_6H_4\cdot CH_3$	Toluidino
22	—$NH\cdot CO\cdot NH_2$	Ureido
23	—$NH\cdot C(:NH)\cdot NH_2$	Guanidino
24	—NH·OH	Hydroxylamino
25	—$NH\cdot NH_2$	Hydrazino
26	—NH·NH—	Hydrazo
27	—N:N—	Azo
28	$\cdot N{:}N]^+X$	Diazonium, Diazo (X = OH, Cl, etc.)
29	$-\underset{O}{\overset{}{N{=}N}}-$ $\left(\begin{array}{c}-N{=}N-\\ \downarrow\\ O\end{array}\right)$	Azoxy
30	—As:As—	Arseno
31	—NH·N:N— (*open*)	Diazoamino
32	—NH·N:N— (*cyclic*)	Azimino
33	—CH_3	Methyl
34	—CH_2OH	Hydroxymethyl, Methylol
35	—C_2H_5	Ethyl
36	—$CH_2\cdot CH_2\cdot CH_3$	*n*-Propyl
37	—$CH(CH_3)_2$	Isopropyl
38	—$CH_2\cdot CH_2\cdot CH_2\cdot CH_3$	*n*-Butyl
39	—$CH_3\cdot \underset{\vert}{C}H\cdot CH_2\cdot CH_3$	*sec.*-Butyl
40	—$CH_2\cdot CH(CH_3)_2$	Isobutyl
41	—$C(CH_3)_3$	*tert.*-Butyl
42	—$CH_2\cdot[CH_2]_3\cdot CH_3$	*n*-Amyl
43	—$CH_3\cdot \underset{\vert}{C}H\cdot CH_2\cdot CH_2\cdot CH_3$	*sec.*-Amyl
44	—$CH_2\cdot CH_2\cdot CH(CH_3)_2$	Isoamyl
45	—$CH_3\cdot \underset{\vert}{C}H\cdot CH(CH_3)_2$	*sec.*-Isoamyl
46	—$CH_2\cdot CH\begin{array}{l}\diagup CH_3\\ \diagdown C_2H_5\end{array}$	*active* Amyl
47	—$C\begin{array}{l}\diagup CH_3\\ -C_2H_5\\ \diagdown CH_3\end{array}$	*tert.*-Amyl
48	—$CH(CH_2\cdot CH_3)_2$	Diethylmethyl
49	—$CH_2\cdot[CH_2]_4\cdot CH_3$	*n*-Hexyl
50	—$CH_2\cdot[CH_2]_2\cdot CH(CH_3)_2$	Isohexyl
51	—$CH_2\cdot[CH_2]_5\cdot CH_3$	*n*-Heptyl, Oenanthyl
52	—$CH_2\cdot[CH_2]_3\cdot CH(CH_3)_2$	Isoheptyl
53	—$CH_2\cdot[CH_2]_6\cdot CH_3$	Octyl, Capryl
54	—$CH_2\cdot[CH_2]_7\cdot CH_3$	Nonyl
55	—$CH_2\cdot[CH_2]_8\cdot CH_3$	Decyl
56	—$CH_2\cdot[CH_2]_9\cdot CH_3$	Undecyl
57	—$CH_2\cdot[CH_2]_{10}\cdot CH_3$	Dodecyl
58	—$CH_2\cdot[CH_2]_{11}\cdot CH_3$	Tridecyl
59	—$CH_2\cdot[CH_2]_{12}\cdot CH_3$	Tetradecyl
60	—$CH_2\cdot[CH_2]_{13}\cdot CH_3$	Pentadecyl
61	—$CH_2\cdot[CH_2]_{14}\cdot CH_3$	Cetyl, Hexadecyl
62	—$CH_2\cdot[CH_2]_{15}\cdot CH_3$	Heptadecyl
63	—$CH_2\cdot[CH_2]_{16}\cdot CH_3$	Octadecyl
64	—$CH_2\cdot[CH_2]_{18}\cdot CH_3$	Eicosyl
65	—$CH_2\cdot[CH_2]_{19}\cdot CH_3$	Heneicosyl
66	—$CH_2\cdot[CH_2]_{24}\cdot CH_3$	Ceryl
67	—$CH_2\cdot[CH_2]_{28}\cdot CH_3$	Myricyl, Melissyl
68	—$CH\begin{array}{l}\diagup CH_2\\ \vert\\ \diagdown CH_2\end{array}$	Cyclopropyl (*followed by* Cyclobutyl, Cyclopentyl, Cyclohexyl, Cycloheptyl (Suberyl) *in that order*)
69	—$CH{:}CH_2$	Vinyl
70	—$CH{:}CH\cdot CH_3$	Propenyl
71	—$C({:}CH_2)\cdot CH_3$	Isopropenyl
72	—$CH_2\cdot CH{:}CH_2$	Allyl
73	—$CH{:}CH\cdot CH_2\cdot CH_3$	α-Butenyl
74	—$CH_2\cdot CH{:}CH\cdot CH_3$	β-Butenyl, Crotyl
75	—$CH_2\cdot CH_2\cdot CH{:}CH_2$	γ-Butenyl, Allylomethyl
76	—$CH{:}CH\cdot CH_2\cdot CH_2\cdot CH_3$	α- (β- etc.) Pentenyl
77	—$CH{:}CH\cdot[CH_2]_3\cdot CH_3$	α- (β- etc.) Hexenyl
78	—$CH_2\cdot[CH_2]_7\cdot CH{:}CH\cdot[CH_2]_7\cdot CH_3$	Octadecenyl
79	—C⋮CH	Ethynyl, Acetylenyl
80	—$CH_2\cdot C$⋮CH	Propargyl
81	—C_6H_5	Phenyl
82	—$C_6H_4\cdot CH_3$	Tolyl
83	—$CH_2\cdot C_6H_5$	Benzyl
84	—$CH_2\cdot C_6H_4\cdot OH$ (-*o*)	Salicyl
85	—$CH_2\cdot C_6H_4\cdot OCH_3$ (-*p*)	Anisyl
86	—$CH_2\cdot CH_2\cdot C_6H_5$	Phenylethyl
87	—$CH_2\cdot C_6H_4\cdot CH_3$	Xylyl
88	—$CH_2\cdot C_{10}H_7$	Menaphthyl
89	—$C_6H_4\cdot CH(CH_3)_2$	Cumyl
90	—$C_6H_2(CH_3)_3$ (1 : 2 : 4)	ψ-Cumyl
91	—$C_6H_2(CH_3)_3$ (1 : 3 : 5)	Mesityl
92	—$CH{:}CH\cdot C_6H_5$	Styryl
93	—$CH_2\cdot CH{:}CH\cdot C_6H_5$	Cinnamyl
94	—$C_{10}H_7$	Naphthyl
95	—$C_6H_4\cdot C_6H_5$	Diphenylyl, Xenyl
96	—$CH(C_6H_5)_2$	Benzhydryl, Diphenylmethyl
97	—$C_{14}H_9$	Anthryl, anthranyl
98	—$C_{14}H_9$	Phenanthryl
99	—$C(C_6H_5)_3$	Triphenylmethyl
100	—$CH_2\cdot CH_2$—	Ethylene, Dimethylene
101	—$CH(CH_3)\cdot CH_2$—	Propylene
102	—$CH_2\cdot CH_2\cdot CH_2$—	Trimethylene
103	—$CH_2\cdot CH_2\cdot CH_2\cdot CH_2$—	Tetramethylene
104	—$C(CH_3)_2\cdot CH_2$—	Isobutylene
105	—$CH_2\cdot[CH_2]_3\cdot CH_2$—	Pentamethylene
106	—$CH_2[\cdot CH_2]_4\cdot CH_2$—	Hexamethylene
107	—$CH_2\cdot[CH_2]_5\cdot CH_2$—	Heptamethylene
108	—$CH_2\cdot[CH_2]_6\cdot CH_2$—	Octamethylene
109	—CH:CH—	Vinylene
110	—C_6H_4—	Phenylene
111	—$C_6H_3(CH_3)$—	Tolylene
112	—CH_2—	Methylene
113	=$CH\cdot CH_3$	Ethylidene
114	=$CH\cdot CH_2\cdot CH_3$	Propylidene
115	=$C(CH_3)_2$	Isopropylidene
116	=$CH\cdot CH_2\cdot CH_2\cdot CH_3$	Butylidene

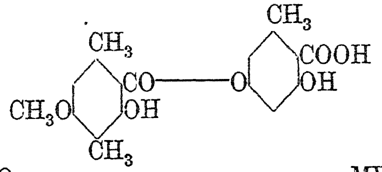
117	=CH·CH(CH$_3$)$_2$	Isobutylidene
118		Cyclohexylidene
119	=C:CH$_2$	Vinylidene
120	=CH·CH:CH$_2$	Allylidene
121	CH$_3$·CH:CH·CH=	Crotylidene
122	=CH·C$_6$H$_5$	Benzylidene
123	=CH·C$_6$H$_4$·OH (-*o*)	Salicylidene
124	=CH·C$_6$H$_4$·OCH$_3$ (-*p*)	Anisylidene
125	=CH·C$_6$H$_4$·CH(CH$_3$)$_2$ (-*p*)	Cuminylidene
126	=CH·CH:CH·C$_6$H$_5$	Cinnamylidene
127	—CH$_2$·CO·CH$_3$	Acetonyl
128	—CH$_2$·CO·C$_6$H$_5$	Phenacyl
129	—CH$_2$·CO·C$_6$H$_4$·CH$_3$	Tolacyl
130	C$_6$H$_5$·CH·CO·C$_6$H$_5$	Desyl
131	—CHO	Aldehydo, Formyl
132	≡CH	Methynyl
133	—CO·CH$_3$	Acetyl, Aceto
134	—CO·CH$_2$·CH$_3$	Propionyl
135	—CO·CH$_2$·CH$_2$·CH$_3$	Butyryl
136	—CO·CH(CH$_3$)$_2$	Isobutyryl
137	—CO·CH$_2$·[CH$_2$]$_2$·CH$_3$	Valeryl
138	—CO·CH$_2$·CH(CH$_3$)$_2$	Isovaleryl
139	—CO·CH$_2$·[CH$_2$]$_3$·CH$_3$	Caproyl
140	—CO·CH$_2$·[CH$_2$]$_{13}$·CH$_3$	Palmityl
141	—CO·CH$_2$·[CH$_2$]$_{15}$·CH$_3$	Stearyl
142	—CO·[CH$_2$]$_7$·CH:CH·[CH$_2$]$_7$·CH$_3$	Oleyl
143	—CO·COOH	Oxalo
144	—CO·C$_6$H$_5$	Benzoyl
145	—CO·C$_6$H$_4$·OH (-*o*)	Salicyloyl
146	—CO·C$_6$H$_4$·OCH$_3$ (-*p*)	Anisoyl
147	—CO·CH$_2$·C$_6$H$_5$	Phenylacetyl
148	—CO·C$_6$H$_4$·CH$_3$	Toluyl
149	—CO·CH:CH·C$_6$H$_5$	Cinnamoyl
150	—CO·C$_{10}$H$_7$	Naphthoyl
151	—CO·CO—	Oxalyl
152	—CO·CH$_2$·CO—	Malonyl
153	—CO·CH$_2$·CH$_2$·CO—	Succinyl
154	—CO·C$_6$H$_4$·CO—	Phthaloyl, Isophthaloyl, Terephthaloyl
155	—COOH (—CO·OCH$_3$, —CO·OC$_2$H$_5$, etc.)	Carboxy, (Carbomethoxy, Carbethoxy, etc.)
156	—CO·NH$_2$	Carbamyl
157	>CO	Carbonyl
158	—C(:NH)·NH$_2$	Guanyl
159	—CN	Cyano
160	—CO·CH$_2$NH$_2$	Glycyl
161	—CO·CH(NH$_2$)·CH$_3$	α-Alanyl
162	—CO·CH$_2$·CH$_2$NH$_2$	β-Alanyl
163	—CO·CH(NH$_2$)·CH(CH$_3$)$_2$	Valyl
164	—CO·CH(NH$_2$)·CH$_2$·CH(CH$_3$)$_2$	Leucyl
165	—CO·CH$_2$·NH·CO·C$_6$H$_5$	Hippuryl
166	—C$_4$H$_3$O	Furyl
167	—C$_4$H$_3$S	Thienyl
168	—CH$_2$·C$_4$H$_3$O	Furfuryl
169	=CH·C$_4$H$_3$O	Furfurylidene
170	—CO·C$_4$H$_3$O	Furoyl, Pyromucyl
171	—C$_4$H$_3$NH	Pyrryl
172	—C$_5$H$_4$N	Pyridyl
173	—C$_5$H$_{10}$N	Piperidyl
174	—NC$_5$H$_{10}$	Piperidino

DICTIONARY OF ORGANIC COMPOUNDS

O

Obaculactone.
See Limonin.

Obtusatic Acid (*Ramalic acid, protocetraric acid*)



$C_{18}H_{18}O_7$ MW, 346

Occurs in the lichen *Ramalina obtusata*, Arnold. Cryst. from Me_2CO or EtOH. M.p. 203° decomp. Mod. sol. Me_2CO. Spar. sol. Et_2O, $CHCl_3$, C_6H_6. $FeCl_3 \longrightarrow$ red col. with EtOH sol.

Di-Me ether Me ester: $C_{21}H_{24}O_7$. MW, 388. M.p. 126°.

Diacetyl: m.p. 175° decomp. *Me ester*: m.p. 142°.

Asahina, Fuzikawa, *Chem. Abstracts*, 1934, **28**, 2397; *Ber.*, 1932, **65**, 580.
Koller *et al.*, *Monatsh.*, 1932, **61**, 286; 1933, **62**, 241; 1934, **64**, 3.

Obtusilic Acid (3-*Decenoic acid*, 3-*decylenic acid*, 3-*nonene*-1-*carboxylic acid*)

$CH_3 \cdot [CH_2]_4 \cdot CH:CH \cdot [CH_2]_2 \cdot COOH$

$C_{10}H_{18}O_2$ MW, 170

Constituent of seed oil of *Lindera obtusiloba*. B.p. 148–50°/13 mm. D_4^{20} 0·9197. n_D^{20} 1·4497.

p-*Bromophenacyl ester*: m.p. 43·3°.

Komori, Ueno, *Bull. Chem. Soc. Japan*, 1937, **12**, 226, 433.

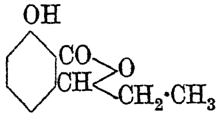
Ochotensine

$C_{21}H_{21}O_4N$ MW, 351

Alkaloid of *Corydalis ochotensis*, Turcz. M.p. 252°. $[\alpha]_D^{23} + 63·9°$ in 0·1*N*/HCl, $[\alpha]_D^{24} + 51·7°$ in $CHCl_3$. Spar. sol. Et_2O. Phenolic.

Manske, *Chem. Abstracts*, 1940, **34**, 4070.

Ochracin (*Mellein*)



$C_{10}H_{10}O_3$ MW, 178

Fermentation product of the mould *Aspergillus ochraceus*. M.p. 58–58·5°. $FeCl_3 \longrightarrow$ violet col. $[\alpha]_D - 124·86°$ ($[\alpha]_D^{12} - 108·15°$ in $CHCl_3$).

Acetyl deriv.: m.p. 126–7°.

Benzoyl deriv.: m.p. 101–2°.

Me ether: $C_{11}H_{12}O_3$. MW, 192. M.p. 88–9°.

Yabuta, Sumika, *Brit. Chem. Abstracts*, 1935, **A**, 619.

Ochrobirine

$C_{20}H_{19}O_6N$ MW, 369

Alkaloid of *Corydalis lutea*. M.p. 138–9°. $[\alpha]_D^{21} + 35·9°$ in $CHCl_3$.

Acetyl deriv.: m.p. 177°.

Manske, *Chem. Abstracts*, 1939, **33**, 6323.

Ocimene (2 : 6-*Dimethyl*-1 : 5 : 7-*octatriene*)

$CH_2:CH \cdot C(CH_3):CH \cdot CH_2 \cdot CH_2 \cdot C(CH_3):CH_2$

$C_{10}H_{16}$ MW, 136

Occurs in leaves of *Litsea Zeylanica*, C. & T., *Boronia dentigeroides*, Cheel, and *Ocimum basilicum*, L. B.p. 176–8° decomp., 81°/30 mm., 73–4°/21 mm. D_4^{21} 0·799. n_D^{18} 1·4857. Prolonged heating $\longrightarrow$ allo-ocimene (*q.v.*).

Romburg, *Chem. Zentr.*, 1901, I, 1006.
Penfold, *Chem. Abstracts*, 1929, **23**, 3303.
Enklaar, *Rec. trav. chim.*, 1926, **45**, 337.

Octabromoacetylacetone (*Phlorobromin, octabromopentandione*-2 : 4)

$CBr_3 \cdot CO \cdot CBr_2 \cdot CO \cdot CBr_3$

$C_5O_2Br_8$ MW, 732

Needles from $CHCl_3$. M.p. 154–5°. Mod. sol. Et_2O, $CHCl_3$, C_6H_6, AcOH. Spar. sol. ligroin. Insol. H_2O. EtOH $\longrightarrow$ $CBr_3 \cdot CO \cdot CHBr_2$. $NH_3 \longrightarrow CBr_3 \cdot CO \cdot NH_2$.

Benedikt, *Ann.*, 1877, **189**, 165.
Zincke, Kegel, *Ber.*, 1890, **23**, 1717.

Octachloroacetylacetone (*Octachloropentandione*-2 : 4)

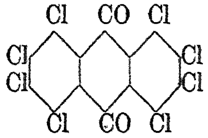
$CCl_3 \cdot CO \cdot CCl_2 \cdot CO \cdot CCl_3$

$C_5O_2Cl_8$ MW, 376

Needles or prisms from ligroin. M.p. 42–3°. B.p. 165–8°/30–2 mm. Heat with $H_2O \longrightarrow CCl_3 \cdot COOH + CCl_3 \cdot CO \cdot CHCl_2$.

Zincke, Kegel, *Ber.*, 1890, **23**, 240.

Octachloroanthraquinone

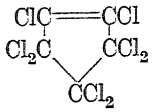


$C_{14}O_2Cl_8$ MW, 484

Yellow needles. Does not melt below 360°.

Eckert, *J. prakt. Chem.*, 1921, **102**, 361.

Octachlorocyclopentene (*Perchlorocyclopentene*)

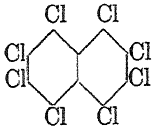


C_5Cl_8 MW, 344

Needles. M.p. 39°. B.p. 283°/760 mm.

Prins, *Rec. trav. chim.*, 1932, **51**, 1068.

Octachloronaphthalene



$C_{10}Cl_8$ MW, 404

Cryst. from C_6H_6–CCl_4. M.p. 197·5–198°. B.p. 440–2°/7·4 mm., 258–60°/2·5 mm., 246–50°/0·5 mm.

Schwemberger, Gordon, *Chem. Zentr.*, 1935, II, 514; *Chem. Abstracts*, 1933, **27**, 2439.

Octacosane

$CH_3\cdot[CH_2]_{26}\cdot CH_3$

$C_{28}H_{58}$ MW, 394

M.p. 64–5° (62°). B.p. 278°/15 mm., 224°/1·1 mm. n_D^{65} 1·43539.

Levene, West, Scheer, *J. Biol. Chem.*, 1915, **20**, 521.
Hildebrand, Wachter, *J. Am. Chem. Soc.*, 1929, **51**, 2487.
Mazee, *Rec. trav. chim.*, 1948, **67**, 197.

Octacosane-1 : 28-dicarboxylic Acid

$HOOC\cdot CH_2\cdot[CH_2]_{26}\cdot CH_2\cdot COOH$

$C_{30}H_{58}O_4$ MW, 482

M.p. 108° (123–5°).
Di-Et ester: $C_{34}H_{66}O_4$. MW, 538. M.p. 74°.

Fairweather, *Chem. Abstracts*, 1927, **21**, 3182.
Ruzicka, Brugger, Seidel, Schinz, *Helv. Chim. Acta*, 1928, **11**, 496.

Octacosanic Acid (*Octacosanoic acid, octacosoic acid*)

$CH_3\cdot[CH_2]_{25}\cdot CH_2\cdot COOH$

$C_{28}H_{56}O_2$ MW, 424

M.p. 90·3–90·5° (89°). n_D^{100} 1·4313.
Et ester: $C_{30}H_{60}O_2$. MW, 452. M.p. 64·8–65°.

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.
Holde, Bleyberg, Vohrer, *Chem. Abstracts*, 1931, **25**, 189.

Octacosanol-1.
See Octacosyl Alcohol.

Octacosyl Alcohol (*Octacosanol*-1)

$CH_3\cdot[CH_2]_{26}\cdot CH_2OH$

$C_{28}H_{58}O$ MW, 410

M.p. 83·2–83·4° (82·9–83·1°).

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.
Pollard, Chibnall, Piper, *Biochem. J.*, 1933, **27**, 1889.

Octacosyl iodide (1-*Iodo*-n-*octacosane*)

$CH_3\cdot[CH_2]_{26}\cdot CH_2I$

$C_{28}H_{57}I$ MW, 520

M.p. 62·8–63·2°.

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.

8 : 10-Octadecadienic Acid

$CH_3\cdot[CH_2]_5\cdot CH{:}CH\cdot CH{:}CH\cdot[CH_2]_7\cdot COOH$

$C_{18}H_{32}O_2$ MW, 280

Me ester: $C_{19}H_{34}O_2$. MW, 294. Liq. B.p. 195–6°/5 mm.
Et ester: $C_{20}H_{36}O_2$. MW, 308. Liq. B.p. 215–16°/5 mm.

Priester, U.S.P., 2,156,737, (*Chem. Abstracts*, 1939, **33**, 6350).
Kartha, Menon, *Chem. Abstracts*, 1943, **37**, 4361.

8 : 11-Octadecadienic Acid.
See Linoleic Acid.

9 : 12-Octadecadienol-1.
See Linoleyl Alcohol.

Octadecamethyleneimine

$$\begin{matrix} CH_2\cdot[CH_2]_7\cdot CH_2 \\ CH_2\cdot[CH_2]_7\cdot CH_2 \end{matrix}\!\!>NH$$

$C_{18}H_{37}N$ MW, 267

Cryst. M.p. 66°. pK 9·33.

Ruzicka, Kobelt, Hafliger, Prelog, *Helv. Chim. Acta*, 1949, **32**, 544.

Octadecane

$CH_3\cdot[CH_2]_{16}\cdot CH_3$

$C_{18}H_{38}$ MW, 254

F.p. 27·5°. (i) Transparent cryst. from Me_2CO. M.p. 27·6°. (ii) Opaque cryst. M.p. 28·02°. B.p. 305–7°, 185–7°/20 mm., 180–3°/15 mm. D_4^{28} 0·7768.

Ueno, *Chem. Abstracts*, 1930, **24**, 4948.
Schrauth, Schenck, Stickdorn, *Ber.*, 1931, **64**, 1317.
Matsui, Arakawa, *Chem. Abstracts*, 1932, **26**, 5264.
Carey, Smith, *J. Chem. Soc.*, 1933, 346.

Octadecane-1 : 18-dicarboxylic Acid

$HOOC\cdot CH_2\cdot[CH_2]_{16}\cdot CH_2\cdot COOH$

$C_{20}H_{38}O_4$ MW, 342

M.p. 124–5° (123°).
Di-Me ester: $C_{22}H_{42}O_4$. MW, 370. M.p. 65·5–66°. B.p. 233–4°/2 mm.

Di-Et ester: $C_{24}H_{46}O_4$. MW, 398. M.p. 54·5–55°. B.p. 230–2°/2 mm.

Chuit, Hausser, *Helv. Chim. Acta*, 1929, **12**, 856.

1 : 2-Octadecanediol (1 : 2-*Dihydroxyoctadecane*)

$CH_3{\cdot}[CH_2]_{15}{\cdot}CH(OH){\cdot}CH_2OH$

$C_{18}H_{38}O_2$ MW, 286

Cryst. from MeOH–ligroin. M.p. 79–79·5°.
Diacetyl: cryst. from MeOH. M.p. 40°.

Niemann, Wagner, *J. Org. Chem.*, 1942, **7**, 227.
Swern, Billen, Scanlan, *J. Am. Chem. Soc.*, 1946, **68**, 1504.

1 : 18-Octadecanediol (1 : 18-*Dihydroxyoctadecane*)

$HOCH_2{\cdot}[CH_2]_{16}{\cdot}CH_2OH$

$C_{18}H_{38}O_2$ MW, 286

Colourless needles from dioxan or C_6H_6. M.p. 97–8°.

Bennett, Gudgeon, *J. Chem. Soc.*, 1938, 1679.
Drake *et al.*, *J. Am. Chem. Soc.*, 1941, **63**, 617.

Octadecanol-1.
See Octadecyl Alcohol.

Octadecanol-3.
See Ethylpentadecylcarbinol.

Octadecanone.
See Ethyl pentadecyl Ketone *and* Methyl hexadecyl Ketone.

1-Octadecene (1-*Octadecylene*)

$CH_3{\cdot}[CH_2]_{15}{\cdot}CH{:}CH_2$

$C_{18}H_{36}$ MW, 252

M.p. 17·5°. B.p. 144–6°/8 mm. n_D^{20} 1·4448, n_D^{30} 1·4412.

Niemann, Wagner, *J. Org. Chem.*, 1942, **7**, 227.
Asinger, *Ber.*, 1942, **75**, 1247.

Δ^9-Octadecenyl Alcohol (9-*Octadecenol*-1)

$CH_3{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}CH_2OH$

$C_{18}H_{36}O$ MW, 268

Cis: Oleyl alcohol, oleic alcohol.
Occurs in fish oils. B.p. 205–10°/15 mm. D_4^{20} 0·8489. n_D^{20} 1·4607. Conc. sulphuric acid ⟶ sulphuric esters possessing powerful detergent, wetting, and emulsifying properties.
Acetyl: D_4^{20} 0·8704. n_D^{20} 1·4515.

Reid, Cockerille, Meyer, Cox, Ruhoff, *Organic Syntheses*, 1935, XV, 72.
Noller, Bannerot, *J. Am. Chem. Soc.*, 1934, **56**, 1563.
Hirose, *Chem. Abstracts*, 1930, **24**, 2321.
Toyama, *Chem. Abstracts*, 1924, **18**, 1270.
Hansley, *Ind. Eng. Chem.*, 1947, **39**, 55.
Nystrom, Brown, *J. Am. Chem. Soc.*, 1947, **69**, 1197.

Trans: elaidyl alcohol, elaidic alcohol.
Constituent of sperm oil. M.p. 36–7° (34°). B.p. 216°/18 mm. D^{40} 0·8388. n_D^{40} 1·4552.
Phenylurethane: m.p. 55°.
β-*Naphthylurethane*: m.p. 71°.

Böeseken, Belinfante, *Rec. trav. chim.*, 1926, **45**, 915.
André, Francois, *Compt. rend.*, 1927, **185**, 279.
Toyama, *Chem. Umschau*, 1924, **31**, 13.

Octadecyl Alcohol (1-*Hydroxyoctadecane*, *stearyl alcohol*, *octadecanol*-1)

$CH_3{\cdot}[CH_2]_{16}{\cdot}CH_2OH$

$C_{18}H_{38}O$ MW, 270

Leaflets from EtOH. F.p. 57·95°. M.p. 59·4–59·8°. B.p. 210°/15 mm., 195–205°/0·2 mm. D_4^{59} 0·8124. Conc. H_2SO_4 ⟶ sulphuric ester possessing detergent, wetting, and emulsifying properties.
Acetyl: (i) f.p. 30·25°. (ii) M.p. 32·85°.
p-*Nitrobenzoyl*: cryst. M.p. 65°.
2 : 4-*Dinitrobenzoyl*: cryst. M.p. 76·5°.
Phenylurethane: needles from EtOH. M.p. 79–80°.
o-*Nitrophenylurethane*: m.p. 70°.
m-*Nitrophenylurethane*: m.p. 77°.
3 : 5-*Dinitrophenylurethane*: m.p. 88°.
Me ether: $C_{19}H_{40}O$. MW, 284. Laminæ from Et_2O–MeOH. M.p. 30–1°.
α-*Glyceryl ether*: *see* Batyl Alcohol.
β-*Glyceryl ether*: $C_{21}H_{44}O_3$. MW, 344. Needles from EtOH. M.p. 62–3°. *Di-phenylurethane*: m.p. 83–4°.

Jantzen, Tiedcke, *J. prakt. Chem.*, 1930, **127**, 277.
Schrauth, Schlenck, Stickdorn, *Ber.*, 1931, **64**, 1318.
Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2510.
Phillips, Mumford, *J. Chem. Soc.*, 1932, 904; 1933, 235.
Tsujimoto, *Chem. Abstracts*, 1921, **15**, 2006.
Nystrom, Brown, *J. Am. Chem. Soc.*, 1947, **69**, 2548.
Meiser, *Chem. Abstracts*, 1948, **42**, 221.

Octadecylamine (*Stearylamine*)

$CH_3{\cdot}[CH_2]_{16}{\cdot}CH_2NH_2$

$C_{18}H_{39}N$ MW, 269

Liq. B.p. 172–3°/2·5 mm.
Acetate: long needles from EtOH. M.p. 84–5°.

Ralston, *Oil and Soap*, 1940, **17**, 89, (*Chem. Abstracts*, 1940, **34**, 3674).
Harwood, Ralston, Selby, *J. Am. Chem. Soc.*, 1941, **63**, 1916.

Octadecylbenzene (1-*Phenyloctadecane*)

$CH_3{\cdot}[CH_2]_{17}{\cdot}C_6H_5$

$C_{24}H_{42}$ MW, 330

M.p. 36°. B.p. 249°/15 mm., 147°/0 mm. D^{36} 0·8510. n_D^{36} 1·4751.

Krafft, *Ber.*, 1886, **19**, 2984.
Mikeska, Smith, Lieber, *J. Org. Chem.*, 1938, **2**, 500.
Gilman, Meals, *J. Org. Chem.*, 1943, **8**, 126.

Octadecyl bromide (1-*Bromo-octadecane*)

$CH_3\cdot[CH_2]_{16}\cdot CH_2Br$

$C_{18}H_{37}Br$ MW, 333

Cryst. from EtOH. M.p. 28·5°. B.p. 168–9°/1·5 mm. Sol. EtOH, AcOEt, pet. ether.

Oskerko, *Chem. Zentr.*, 1914, II, 1264.
Seidel, Engelfried, *Ber.*, 1936, **69**, 2567.

Octadecyl chloride (1-*Chloro-octadecane*)

$CH_3\cdot[CH_2]_{16}\cdot CH_2Cl$

$C_{18}H_{37}Cl$ MW, 288·5

B.p. 185–90°/15 mm. D^{20} 0·9041.

Mabery, *Am. Chem. J.*, 1902, **28**, 165.

Octadecylene.
See Octadecene.

Δ^2-Octadecylenic Acid

$CH_3\cdot[CH_2]_{13}\cdot CH{:}CH\cdot CH_2\cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

Cryst. from AcOH. M.p. 56–7°.
Me ester: $C_{19}H_{36}O_2$. MW, 296. M.p. 36°.

Eckert, Halla, *Monatsh.*, 1913, **34**, 1815.

Δ^3-Octadecylenic Acid

$CH_3\cdot[CH_2]_{12}\cdot CH{:}CH\cdot[CH_2]_2\cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

M.p. 52–3°.

Eckert, Halla, *Monatsh.*, 1913, **34**, 1815.

Δ^5-Octadecylenic Acid.
See Petroselic Acid.

Δ^8-Octadecylenic Acid.
See Elaidic Acid *and* Oleic Acid.

Δ^{10}-Octadecylenic Acid

$CH_3\cdot[CH_2]_5\cdot CH{:}CH\cdot[CH_2]_9\cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

F.p. 6–8°.

Fokin, *Chem. Abstracts*, 1912, **6**, 2409.

Δ^{11}-Octadecylenic Acid

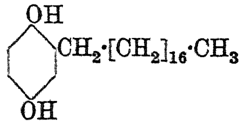
$CH_3\cdot[CH_2]_4\cdot CH{:}CH\cdot[CH_2]_{10}\cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

F.p. 36–8°. M.p. 34–6°.

Fokin, *Chem. Abstracts*, 1912, **6**, 2409.

Octadecylhydroquinone (*Octadecylquinol, 2 : 5-dihydroxyoctadecylbenzene*)



$C_{24}H_{42}O_2$ MW, 362

Cryst. from pet. ether. M.p. 100·5°. Ag_2O ⟶ octadecylbenzoquinone. M.p. 76°.
Di-Me ether: 2 : 5-dimethoxyoctadecylbenzene. B.p. 188°/0·2 mm.
Di-Et ether: 2 : 5-diethoxyoctadecylbenzene. B.p. 201°/0·06 mm.

Cook, Heilbron, Lewis, *J. Chem. Soc.*, 1942, 660.

Octadecylic Acid.
See Stearic Acid.

Octadecyl iodide (1-*Iodo-octadecane*)

$CH_3\cdot[CH_2]_{16}\cdot CH_2I$

$C_{18}H_{37}I$ MW, 380

M.p. 34·5–35° (32·94°). B.p. 160–70°/0·5 mm. Spar. sol. EtOH.

Jantzen, Tiedcke, *J. prakt. Chem.*, 1930, **127**, 277.
Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2510.
Carey, Smith, *J. Chem. Soc.*, 1933, 346.

Octadecylmalonic Acid (*Nonadecane*-1 : 1-*dicarboxylic acid*)

$CH_3\cdot[CH_2]_{17}\cdot CH{<}^{COOH}_{COOH}$

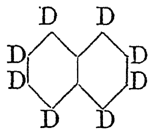
$C_{21}H_{40}O_4$ MW, 356

Cryst. from AcOH. M.p. 109–10°. Sol. Et_2O. Insol. pet. ether.
Amide: $C_{21}H_{41}O_3N$. MW, 355. Cryst. from AcOH. M.p. 126° with loss of CO_2. Insol. Et_2O.
Mononitrile: 1-cyano-*n*-eicosanic acid. $C_{21}H_{39}O_2N$. MW, 337. Cryst. powder from EtOH. M.p. 88°.

Baczewski, *Monatsh.*, 1896, **17**, 544.
Meyer, Brod, Soyka, *Monatsh.*, 1913, **34**, 1132.

Octadecyne-1.
See Hexadecylacetylene.

Octadeuteronaphthalene



$C_{10}D_8$ MW, 136

Cryst. from MeOH.Aq. M.p. 77·5°.

Clemo, McQuillen, *J. Chem. Soc.*, 1935, 1325.

2 : 4-Octadiene (*Propylpropenylethylene, methylpropylbutadiene*)

$CH_3\cdot CH_2\cdot CH_2\cdot CH{:}CH\cdot CH{:}CH\cdot CH_3$

C_8H_{14} MW, 110

B.p. 133·5–134°. D_4^{25} 0·7427. n_D^{25} 1·4542.

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, **57**, 1607.

2 : 6-Octadiene (*Dicrotyl, propenylethylidene-propane*)

$CH_3 \cdot CH:CH \cdot CH_2 \cdot CH_2 \cdot CH:CH \cdot CH_3$

C_8H_{14} MW, 110

B.p. 117–19°.

Charon, *Ann. chim.*, 1899, **17**, 265.

3 : 5-Octadiene (1 : 4-*Diethylbutadiene, dipropenylethane*)

$CH_3 \cdot CH_2 \cdot CH:CH \cdot CH:CH \cdot CH_2 \cdot CH_3$

C_8H_{14} MW, 110

B.p. 138–40°.

Kaufmann, Schweitzer, *Ber.*, 1922, **55**, 262.

3 : 5-Octadiene-2 : 7-dione (2 : 7-*Diketo-3 : 5-octadiene*, 1 : 4-*diaceto*-1 : 3-*butadiene*)

$CH_3 \cdot CO \cdot CH:CH \cdot CH:CH \cdot CO \cdot CH_3$

$C_8H_{10}O_2$ MW, 138

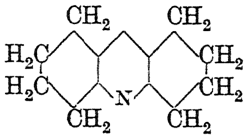
Colourless cryst. M.p. 126–126·5°. Sol. Me_2CO, C_6H_6, MeOH, EtOH, $CHCl_3$, AcOEt, Py. Mod. sol. pet. ether. Insol. H_2O. Light absorption : $\lambda_{max.}$ 276 mμ; log ε = 4·52 (in EtOH).

Dioxime : cryst. from EtOH. M.p. 231°.
Disemicarbazone : cryst. M.p. 249°.

Karrer, Eugster, Perl, *Helv. Chim. Acta*, 1949, **32**, 1014, 1935.

Octafluoropropane.
See Profluorane.

***sym.*-Octahydroacridine**



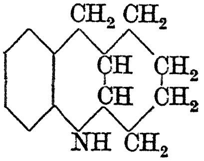
$C_{13}H_{17}N$ MW, 187

Cryst. from ligroin. M.p. 69°. B.p. 175°/17 mm.

B_2,H_2PtCl_6 : m.p. 199–200°.
Picrate : m.p. 195°.
Methiodide : m.p. 159°.

Braun, Petzold, Schultheiss, *Ber.*, 1923, **56**, 1349.

***unsym.*-Octahydroacridine**



$C_{13}H_{17}N$ MW, 187

d-.

(*a*) M.p. 84·5°. $[\alpha]_D$ + 34°. (*b*) M.p. 72°. $[\alpha]_D$ + 22°.

l-.

(*a*) M.p. 85°. $[\alpha]_D$ − 32°. (*b*) M.p. 72°. $[\alpha]_D$ − 24°.

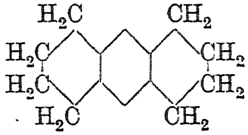
Camphorsulphonate : (*a*) M.p. 183°. (*b*) M.p. 172°.

dl-.

(*a*) M.p. 84°. B.p. 183°/12 mm. *N-Benzoyl* : m.p. 104°. (*b*) Plates from pet. ether. M.p. 72°. *N-Acetyl* : m.p. 136°. *Picrate* : m.p. 175°. *Methiodide* : m.p. 217°.

Braun, Heymons, Manz, *Ber.*, 1931, **64**, 233.
Perkin, Sedgwick, *J. Chem. Soc.*, 1924, **125**, 2448.
Adkins, Coonradt, *J. Am. Chem. Soc.*, 1941, **63**, 1569.

***sym.*-Octahydroanthracene**

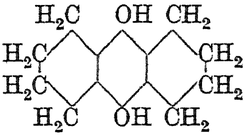


$C_{14}H_{18}$ MW, 186

Leaflets from EtOH. M.p. 78°.

Sugino, Outi, *J. Chem. Soc. Japan*, 1941, **62**, 401, (*Chem. Abstracts*, 1943, **37**, 4726).

***sym.*-Octahydroanthrahydroquinone**

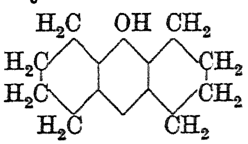


$C_{14}H_{18}O_2$ MW, 218

Cryst. from EtOH. M.p. 242° (234–6°).
Diacetyl : m.p. 224°.

Braun, Bayer, *Ber.*, 1925, **58**, 2680.
Skita, *ibid.*, 2692.

***sym.*-Octahydroanthranol**

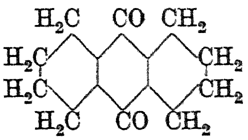


$C_{14}H_{18}O$ MW, 202

Needles from EtOH. M.p. 125°.
Acetyl : m.p. 52°.
Benzoyl : m.p. 129° (128°).

Skita, *Ber.*, 1925, **58**, 2693.
Braun, Bayer, *ibid.*, 2678.

***sym.*-Octahydroanthraquinone**



$C_{14}H_{16}O_2$ MW, 216

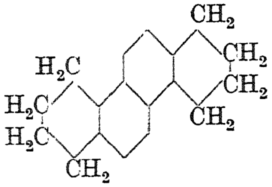
Cis–cis :
Leaflets from MeOH. M.p. 154–5°.

Cis–trans :
Needles from EtOH. M.p. 186°.

Trans–trans :
Leaflets from AcOEt. M.p. 245°.

Alder, Stein, *Ann.*, 1933, **501**, 283.
Skita, *Ber.*, 1925, **58**, 2692.
Braun, Bayer, *ibid.*, 2678.

sym.-Octahydrochrysene

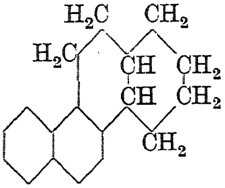


$C_{18}H_{20}$ MW, 236

M.p. 136–8° (138–40°). B.p. 180–1°/0·2 mm.
Picrate : m.p. 139–40°.

Braun, Irmisch, *Ber.*, 1932, **65**, 885.
Cohen, Cook, Hewett, *J. Chem. Soc.*, 1935, 1636.

unsym.-Octahydrochrysene



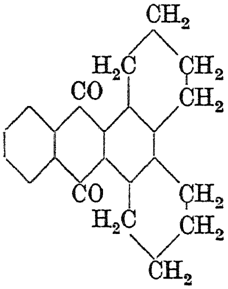
$C_{18}H_{20}$ MW, 236

Cis (?) :
Needles from EtOH. M.p. 78–9°.
Picrate : m.p. 106–106·5°.

Trans (?) :
Needles from MeOH. M.p. 114–114·5°. Forms no picrate.

Cook, Hewett, *J. Chem. Soc.*, 1934, 372.

Octahydro-1 : 2 : 3 : 4-dibenzanthraquinone

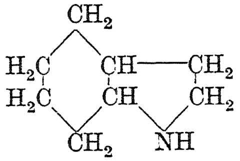


$C_{22}H_{20}O_2$ MW, 316

Yellow cryst. from xylene. M.p. 234°. Conc. H_2SO_4 ⟶ red col.

Cook, Hewett, *J. Chem. Soc.*, 1934, 372.
Bergmann, Eschinazi, Neeman, *J. Org. Chem.*, 1943, **8**, 180.
Barnett, Goodway, Lawrence, *J. Chem. Soc.*, 1935, 1684.

Octahydroindole (*Perhydroindole*)

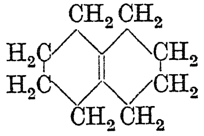


$C_8H_{15}N$ MW, 125

Oil with onion-like odour. B.p. 185·5°/760 mm., 182–3°/720 mm., 64–5°/12 mm. Sol. usual org. solvents. Insol. H_2O. Reacts alkaline. D_4^{20} 0·9472. n_D^{20} 1·4892.
B_2,H_2PtCl_6 : reddish-yellow leaflets from EtOH. M.p. about 172–3°. Very sol. warm EtOH. Insol. cold H_2O.
Benzenesulphonyl : cryst. M.p. 70–1°.
Picrate : needles from EtOH. M.p. 137–8°. Very sol. hot EtOH, $CHCl_3$. Spar. sol. C_6H_6.

Willstätter, Jaquet, *Ber.*, 1918, **51**, 778.
Willstätter, Seitz, Braun, *Ber.*, 1925, **58**, 385.
Adkins, Coonradt, *J. Am. Chem. Soc.*, 1941, **63**, 1563.

1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydronaphthalene (Δ^9-*Octalin*)

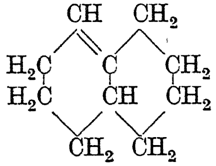


$C_{10}H_{16}$ MW, 136

B.p. 196·5–198·8°/759 mm. D_4^{20} 0·9170. n_D^{20} 1·4996.
Dibromide : small plates from ligroin. M.p. 163–4°.
Nitrosochloride : massive blue prisms from Et_2O. M.p. 91°.

Linstead, Wang, Williams, Errington, *J. Chem. Soc.*, 1937, 1139.

1 : 2 : 3 : 4 : 5 : 6 : 7 : 10-Octahydronaphthalene ($\Delta^{1,9}$-*Octalin*)

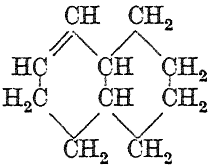


$C_{10}H_{16}$ MW, 136

B.p. 189·5–193·5°/759 mm. $D_4^{20\cdot8}$ 0·9027.
Nitropiperidide : m.p. 179° decomp.
Nitrosochloride : m.p. 127°.

Linstead, Wang, Williams, Errington, *J. Chem. Soc.*, 1937, 1139.
Hückel, Tappe, Legutke, *Ann.*, 1940, **543**, 191.

1 : 2 : 3 : 4 : 5 : 6 : 9 : 10-Octahydronaphthalene (Δ^1-*Octalin*)



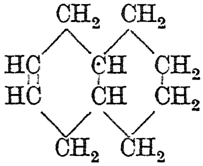
$C_{10}H_{16}$ MW, 136

Trans :

B.p. 185°. $D_4^{15·6}$ 0·8970. $n_{He}^{15·6}$ 1·4872.

Hückel, Naub, *Ann.*, 1933, **502**, 150.
Cf. Borsche, Lange, *Ann.*, 1923, **434**, 225.
Hückel, Tappe, Legutke, *Ann.*, 1940, **543**, 191.

1 : 2 : 3 : 4 : 5 : 8 : 9 : 10-Octahydronaphthalene (Δ^2-*Octalin*, β-*naphthanene*)



$C_{10}H_{16}$ MW, 136

B.p. 197–9°. D^{20} 0·9103. n_D^{20} 1·4941.

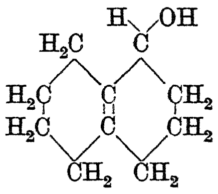
Trans:
M.p. − 14° (− 24°). B.p. 62°/22 mm. D_4^{20} 0·89414, D_4^{17} 0·8956. n_D^{20} 1·48317, n_D^{17} 1·4859. Heat + P_2O_5 ⟶ Δ^9-octalin.

d-Trans : $[\alpha]_D$ + 11·7°. Alk. $KMnO_4$ ⟶ optically impure *l*-cyclohexane-1 : 2-diacetic acid.

l-Trans : b.p. 72°/13 mm. D_4^{20} 0·89414. $[\alpha]_D^{20}$ − 25·2° in EtOH, − 26·3° in cyclohexane, − 27·45° in C_6H_6. Alk. $KMnO_4$ ⟶ optically impure *d*-cyclohexane-1 : 2-diacetic acid.

Hückel, *Ber.*, 1925, **58**, 1451.
Cf. Borsche, Lange, *Ann.*, 1923, **434**, 225.
Hückel, Sowa, *Ber.*, 1941, **74**, 62.
Hückel, Tappe, Legutke, *Ann.*, 1940, **543**, 191.

Octahydro-1-naphthol

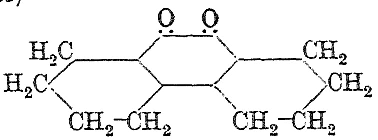


$C_{10}H_{16}O$ MW, 152

Liq. B.p. 133–4°/16 mm. D_4^{16} 1·0038. n_D^{16} 1·5854.
Acetyl : viscous liq. B.p. 131°/12 mm. D_4^{16} 1·0162. n_D^{16} 1·4860.

Burnop, Linstead, *J. Chem. Soc.*, 1940, 720.
Campbell, Harris, *J. Am. Chem. Soc.*, 1941, **63**, 2721.

***sym*.-Octahydrophenanthraquinone** (*Octanthrone*)

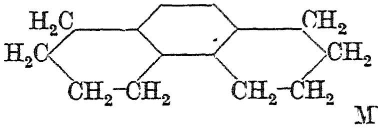


$C_{14}H_{16}O_2$ MW, 216

Red needles from MeOH. M.p. 142°.

Skita, Warnat, *Ber.*, 1925, **58**, 2691.

1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophenanthrene (*Octanthrene*)

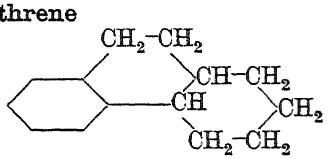


$C_{14}H_{18}$ MW, 186

M.p. 16·7°. B.p. 295°, 172–3°/20 mm., 167·5°/13 mm., 135–6°/6 mm. D^{20} 1·026. n_D^{17} 1·5669.

Schroeter, Müller, Huang, *Ber.*, 1929, **62**, 650.
Schroeter, D.R.P., 352,719, (*Chem. Abstracts*, 1923, **17**, 1246).
Kamp, Mosettig, *J. Am. Chem. Soc.*, 1935, **57**, 1107.
Durland, Adkins, *J. Am. Chem. Soc.*, 1937, **59**, 136.

1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-Octahydrophenanthrene



$C_{14}H_{18}$ MW, 186

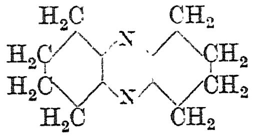
B.p. 159°/15 mm., 135°/9 mm. (135–7°/6·5 mm.). D_4^{32} 0·997325. $n_D^{19·2}$ 1·5527.

Cis :
Colourless liq. B.p. 129°/6 mm., 88–90°/0·1–0·5 mm. D_4^{13} 1·0164, D_4^{25} 1·0072. $n_D^{12·2}$ 1·5586, $n_D^{20·7}$ 1·5549.

Trans :
Colourless needles. M.p. 23–4°. B.p. 94–5°/15 mm. D_4^{20} 1·006, D_4^{25} 0·9840. $n_D^{20·7}$ 1·5528, n_D^{15} 1·5452. $KMnO_4$ + little KOH ⟶ phthalic acid.

Fulton, Robinson, *J. Chem. Soc.*, 1933, 1465.
Bergs, *Ber.*, 1934, **67**, 243.
Kagehira, *Chem. Abstracts*, 1932, **26**, 443.
Cook, Hewett, Robinson, *J. Chem. Soc.*, 1939, 172.
Durland, Adkins, *J. Am. Chem. Soc.*, 1938, **60**, 1504.

1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophenazine

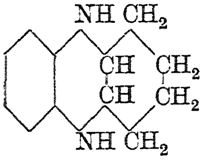


$C_{12}H_{16}N_2$ MW, 188

Cryst. from EtOH.Aq. M.p. 109°.

Clemo, McIlwain, *J. Chem. Soc.*, 1936, 258.
Mousseron, *Bull. soc. chim.*, 1945, **12**, 70.

1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-Octahydrophenazine



$C_{12}H_{16}N_2$ MW, 188

Cis :

Plates from EtOH. M.p. 147°. D^{20} 1·1949, D^{182} 1·0064. n_D^{20} (calc.) 1·6182. Sol. to 18·1% (w/v) in EtOH, to 19·1% in Et_2O at 20°. H + Ni at 140° ⟶ *trans*-isomer.

N : N′-*Dinitroso* : m.p. 109°.

Trans :

Needles from EtOH. M.p. 156°. D^{20} 1·1989, D^{182} 0·9982. n_D^{20} (calc.) 1·6146. Sol. to 23·6% (w/v) in EtOH, to 17·8% in Et_2O at 20°.

N : N′-*Dinitroso* : m.p. 126°.

Clemo, McIlwain, *J. Chem. Soc.*, 1936, 258, 1698.

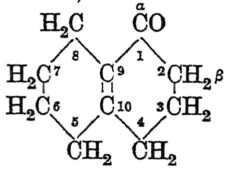
Octahydropyridocoline.
See Norlupinane-A.

Octahydropyrindole.
See Indolizidine.

***n*-Octaldehyde.**
See Caprylic Aldehyde.

Octalin.
See Octahydronaphthalene.

$\Delta^{9,10}$-α-Octalone (1-*Keto*-1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-*octahydronaphthalene*)



$C_{10}H_{14}O$ MW, 150

B.p. 127–8°/10 mm. D_4^{14} 1·000. n_D^{14} 1·4996.

Oxime : m.p. 148°. *Benzoyl deriv.* : m.p. 130·5–131·5°.

Semicarbazone : m.p. 242–3°.

2 : 4-*Dinitrophenylhydrazone* : small dark red tabular cryst. from xylene. M.p. 266·5–267° decomp.

Cook, Lawrence, *J. Chem. Soc.*, 1935, 1638.
Campbell, Harris, *J. Am. Chem. Soc.*, 1941, **63**, 2725; 1942, **64**, 720.

$\Delta^{1,9}$-β-Octalone (2-*Keto*-2 : 3 : 4 : 5 : 6 : 7 : 8 : 10-*octahydronaphthalene*).

Oil. B.p. 140–1°/14 mm. H + Pt ⟶ β-decalone + β-decalol.

Semicarbazone : cream col. prisms from dioxan–EtOH; cryst. from EtOH. M.p. 215°.

2 : 4-*Dinitrophenylhydrazone* : dark red plates from C_6H_6–EtOH. M.p. 171–2°.

Birch, *J. Chem. Soc.*, 1944, 434.
Mannich, Koch, Borkowsky, *Ber.*, 1937, **70**, 358.

$\Delta^{9,10}$-β-Octalone (2-*Keto*-1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-*octahydronaphthalene*).

Colourless oil. B.p. 117°/12 mm.

2 : 4 - *Dinitrophenylhydrazone* : orange-red plates from C_6H_6–EtOH. M.p. 176–7°. Alc. H_2SO_4 ⟶ $\Delta^{1,9}$-isomer.

Birch, *J. Chem. Soc.*, 1946, 597.

Octamethylene.
See Cyclo-octane.

Octamethylene bromide.
See 1 : 8-Dibromo-octane.

Octamethylene chloride.
See 1 : 8-Dichloro-octane.

Octamethylenediamine (1 : 8-*Diamino*-n-*octane*)

$$H_2N{\cdot}CH_2{\cdot}[CH_2]_6{\cdot}CH_2NH_2$$

$C_8H_{20}N_2$ MW, 144

Plates. M.p. 52°. B.p. 240–1° (236–40°, 225–6°), 130–40°/20 mm. Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6, ligroin.

B,2HCl : m.p. 274°.

B,2HAuCl₄ : m.p. 188–9° decomp.

B,(COOH)₂ : m.p. 223°.

Di-picrate : m.p. 180° decomp. (182–3°).

N : N′-*Diacetyl* : m.p. 127–8° (121–2°).

N : N′-*Dibenzoyl* : cryst. from EtOH. M.p. 173°.

Naegeli, Lendorff, *Helv. Chim. Acta*, 1932, **15**, 53.
Neuberg, Neimann, *Z. physiol. Chem.*, 1905, **45**, 117.
Müller, Kindleman, *Ber.*, 1941, **74**, 416.
Hurd, Botteron, *J. Org. Chem.*, 1946, **11**, 213.

Octamethylene Glycol (1 : 8-*Dihydroxy*-n-*octane, octandiol*-1 : 8)

$$HO{\cdot}CH_2{\cdot}[CH_2]_6{\cdot}CH_2OH$$

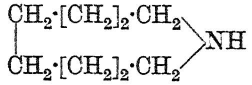
$C_8H_{18}O_2$ MW, 146

Needles from C_6H_6–ligroin. M.p. 63° (60°). B.p. 172°/20 mm., 164°/12 mm., 160–2°/9·5 mm. Sol. EtOH. Spar. sol. H_2O, Et_2O, ligroin.

Diphenylurethane : m.p. 172–172·5°.
Di-Me ether : $C_{10}H_{22}O_2$. MW, 174. B.p. 221°. D^{15} 0·8613. n_D^{15} 1·4257.
Di-n-amyl ether : $C_{18}H_{38}O_2$. MW, 286. B.p. 212°/35 mm.

Lespieau, *Compt. rend.*, 1914, **158**, 1188.
Dionneau, *Bull. soc. chim.*, 1910, **7**, 328.
Bouveault, Blanc, D.R.P., 164,294, (*Chem. Zentr.*, 1905, II, 1701).

Octamethyleneimine



$C_8H_{17}N$ MW, 127

B.p. 90–92°/24 mm. D_4^{21} 0·9021. n_D^{21} 1·4777. pK 9·39.
Picrate : m.p. 135–6°.

Ruzicka, Kobelt, Hafliger, Prelog, *Helv. Chim. Acta*, 1949, **32**, 544.

Octamethylhydrobenzoin.
See Hydroisoduroin.
Octamethylsucrose.
See under Sucrose.
Octanal.
See Caprylic Aldehyde.
Octandiol-1 : 2 (1 : 2-*Dihydroxy*-n-*octane*, n-*hexylethylene glycol*)

$CH_3\cdot[CH_2]_5\cdot CH(OH)\cdot CH_2OH$

$C_8H_{18}O_2$ MW, 146

Cryst. from pet. ether. M.p. 35–7° (30–30·5°). B.p. 135–6°/20 mm. (135–6°/10 mm.). $[\alpha]_D^{17}$ — 4·7° in EtOH.

Späth, Kuffner, Ensfellner, *Ber.*, 1933, **66**, 598.
Swern, Billen, Scanlan, *J. Am. Chem. Soc.*, 1946, **68**, 1505.

Octandiol-1 : 4 (1 : 4-*Dihydroxy*-n-*octane*)

$CH_3\cdot[CH_2]_3\cdot CH(OH)\cdot[CH_2]_2\cdot CH_2OH$

$C_8H_{18}O_2$ MW, 146

Diacetyl : b.p. 127–9°/12 mm. n^{12} 1·4304.

Hinz, Meyer, Schücking, *Reichsamt Wirtschaftsausbau, Chem. Ber.*, 1942, 1043- (*Chem. Abstracts*, 1947, **41**, 6560).

Octandiol-1 : 7 (1 : 7-*Dihydroxy*-n-*octane*)

$CH_3\cdot CH(OH)\cdot[CH_2]_5\cdot CH_2OH$

$C_8H_{18}O_2$ MW, 146

Colourless, viscous liq. B.p. 146–7°/13 mm.
Diacetyl : b.p. 139–42°/13 mm. n^{21} 1·4374.

Hinz, Meyer, Schücking, *Reichsamt Wirtschaftsausbau, Chem. Ber.*, 1942, 1043, (*Chem. Abstracts*, 1947, **41**, 6560).

Octandiol-1 : 8.
See Octamethylene Glycol.

Octandiol-2 : 4 (2 : 4-*Dihydroxy*-n-*octane*)

$CH_3\cdot[CH_2]_3\cdot CH(OH)\cdot CH_2\cdot CH(OH)\cdot CH_3$

$C_8H_{18}O_2$ MW, 146

Oil. B.p. 117–18°/8 mm. D_4^{25} 0·918. n_D^{25} 1·4422.
Di-phenylurethane : m.p. 126–7°.

Stutsman, Adkins, *J. Am. Chem. Soc.*, 1939, **61**, 3306.

Octandiol-2 : 7 (2 : 7-*Dihydroxy*-n-*octane*)

$CH_3\cdot CH(OH)\cdot[CH_2]_4\cdot CH(OH)\cdot CH_3$

$C_8H_{18}O_2$ MW, 146

Oil. B.p. 138–9°/15 mm. Sol. ord. org. solvents.
Di-phenylurethane : m.p. 126°.

Blaise, Koehler, *Bull. soc. chim.*, 1910, **7**, 417.

Octandiol-4 : 5 (4 : 5-*Dihydroxy*-n-*octane*, sym.-*dipropylethylene glycol*)

$CH_3\cdot[CH_2]_2\cdot CH(OH)\cdot CH(OH)\cdot[CH_2]_2\cdot CH_3$

$C_8H_{18}O_2$ MW, 146

dl-.
M.p. 28°. B.p. 109·8–110°/8 mm. $n_D^{24\cdot5}$ 1·4419.
Diacetyl : m.p. 26°. B.p. 110°/5·5 mm. n_D^{20} 1·4283. D_4^{20} 0·9732.
Di-3 : 5-*dinitrobenzoyl* : m.p. 125°.

Meso-.
Leaflets from ligroin. M.p. 123–4°.
Diacetyl : b.p. 100°/5 mm. D_4^{20} 0·9686. n_D^{20} 1·4253.
Di-3 : 5-*dinitrobenzoyl* : cryst. from EtOH–$PhNO_2$. M.p. 200°.

Bouveault, Locquin, *Bull. soc. chim.*, 1906, **35**, 644.
Veibel, *Biochem. Z.*, 1931, **239**, 456.
Young, Levanas, Jasaitis, *J. Am. Chem. Soc.*, 1936, **58**, 2275; 1937, **59**, 404.
Weizmann, Bergmann, Hirshberg, *J. Am. Chem. Soc.*, 1938, **60**, 1532.

Octandione-2 : 3.
See Acetylcaproyl.
Octandione-2 : 4 (*Acetylvalerylmethane*, 2 : 4-*diketo-octane*)

$CH_3\cdot[CH_2]_3\cdot CO\cdot CH_2\cdot CO\cdot CH_3$

$C_8H_{14}O_2$ MW, 142

B.p. 79–83°/20 mm., 64–6°/5 mm. D_4^{20} 0·9233, D_{25}^{25} 0·9218. n_D^{20} 1·4559.

Kutz, Adkins, *J. Am. Chem. Soc.*, 1930, **52**, 4042.
Hurd, Kelso, *J. Am. Chem. Soc.*, 1940, **62**, 2186.

Octandione-2 : 7.
See 1 : 4-Diacetobutane.

Octandione-3 : 4 (*Propionylvaleryl, 3 : 4-diketo-octane*)

$CH_3{\cdot}[CH_2]_3{\cdot}CO{\cdot}CO{\cdot}CH_2{\cdot}CH_3$

$C_8H_{14}O_2$ MW, 142

Dioxime: m.p. about 139–41°.

Fileti, Ponzio, *Gazz. chim. ital.*, 1898, **28**, ii, 264.

Octandione-3 : 5 (*Propionylbutyrylmethane, 3 : 5-diketo-octane*)

$CH_3{\cdot}[CH_2]_2{\cdot}CO{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_3$

$C_8H_{14}O_2$ MW, 142

B.p. 189–90°, 84–6° (75°)/20 mm.

Cu deriv.: blue cryst. from EtOH. M.p. 158·5° (157°).

Powell, Seymour, *J. Am. Chem. Soc.*, 1931, **53**, 1050.
Adams, Hauser, *J. Am. Chem. Soc.*, 1944, **66**, 1222.

Octandione-3 : 6 (sym.-*Dipropionylethane, 3 : 6-diketo-octane*)

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_3$

$C_8H_{14}O_2$ MW, 142

Plates. M.p. 34–5°. B.p. 98°/14 mm.

Dioxime: m.p. 169°.

Blaise, *Compt. rend.*, 1914, **158**, 506.

Octandione-4 : 5.

See Dibutyryl.

***n*-Octane** (*1-Ethylhexane*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_3$

C_8H_{18} MW, 114

F.p. $-$ 56·798 ± 0·008°. B.p. 125·665°. D_4^{20} 0·70252, D_4^{25} 0·69849. n_D^{20} 1·39743, n_D^{25} 1·39505.

Gardiner, Borgstrom, *J. Am. Chem. Soc.*, 1929, **51**, 3375.
Fischer, Klemm, *Z. physik. Chem.*, 1930, **A**, **147**, 275.
Shepard, Henne, Midgley, *J. Am. Chem. Soc.*, 1931, **53**, 1949.
Moldawski, Liwschitz, *Chem. Zentr.*, 1936, I, 758.
Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 725.
Mair, *Bureau of Standards Journal of Research*, 1932, **9**, 463.
Whitmore, Laughlin, *J. Am. Chem. Soc.*, 1933, **55**, 5056 (*Bibl.*).
Maman, *Chem. Zentr.*, 1936, I, 2332.
Forziati *et al.*, *Chem. Abstracts*, 1946, **40**, 4343.
Streiff *et al.*, *Chem. Abstracts*, 1947, **41**, 2384.

Octane-1 : 8-dicarboxylic Acid.

See Sebacic Acid.

Octanoamidoacetaldehyde.

See Penilloaldehyde-K.

Octanol.

See *n*-Octyl Alcohol, *sec.-n*-Octyl Alcohol, Ethyl-*n*-amylcarbinol *and* Propylbutylcarbinol.

5-Octanolone-4.

See Butyroin.

Octanone.

See Ethyl *n*-amyl Ketone, Methyl *n*-hexyl Ketone *and* Propyl butyl Ketone.

Octanthrene.

See 1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophenanthrene.

Octanthrone.

See Octahydrophenanthraquinone.

2 : 4 : 6-Octatrienal

$CH_3{\cdot}[CH{:}CH]_3{\cdot}CHO$

$C_8H_{10}O$ MW, 122

Needles from pet. ether. M.p. 55°. B.p. 57–68°/0·5 mm. Sol. org. solvents. Absorption maximum at 3160 Å in EtOH.

Oxime: yellow. M.p. 186–7° decomp.

Hydrazone: yellow needles from EtOH. Sinters at 153–4°.

2 : 4-*Dinitrophenylhydrazone*: cryst. M.p. 203°.

Azine: orange cryst. from butyl or amyl alcohol. Decomp. at 220–5°.

Fischer, Hultzsch, Flaig, *Ber.*, 1937, **70**, 370.
Kuhn, Hoffer, *Ber.*, 1930, **63**, 2164; 1931, **64**, 1977.
Kuhn, *Angew. Chem.*, 1937, **50**, 703.
Gödde, *Chem. Abstracts*, 1940, **34**, 1970.

1 : 3 : 5-Octatrienedioic Acid (*Hexatriene-1 : 6-dicarboxylic Acid*)

$HOOC{\cdot}[CH{:}CH]_3{\cdot}COOH$

$C_8H_8O_4$ MW, 168

Cryst. M.p. 316–18°.

Di-Me ester: $C_{10}H_{12}O_4$. MW, 196. Cryst. from MeOH. M.p. 172°.

Kuhn, *Angew. Chem.*, 1937, **50**, 703.

2 : 4 : 6-Octatrienol

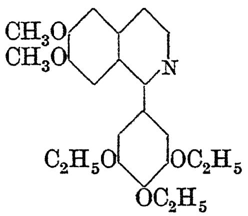
$CH_3{\cdot}[CH{:}CH]_3{\cdot}CH_2OH$

$C_8H_{12}O$ MW, 124

Needles. M.p. 99·5–100·5°. B.p. 95°/12 mm.

Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, **15**, 261, 502.

Octaverine (1-[3 : 4 : 5-*Triethoxyphenyl*]-6 : 7-*dimethoxyisoquinoline*)



$C_{21}H_{23}O_3N$ MW, 337

B,HCl: m.p. 199–200°.

Ellinger, Koschara, Seegar, *Chem. Abstracts*, 1934, **28**, 4476.
Asta A.G., F.P., 760,825, (*Chem. Abstracts*, 1934, **28**, 4178).

1-Octene (1-*Octylene*)

$CH_3\cdot[CH_2]_5\cdot CH{:}CH_2$

C_8H_{16} MW, 112

F.p. — 101·746° (99·77 ± 0·13%, pure). B.p. 121·85–122·15°, 121·15°/742 mm. D_4^{20} 0·7151 (0·71695), D_4^{25} 0·7110. n_D^{20} 1·4087 (1·40765), n_D^{25} 1·4062.

Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 725 (*Bibl.*).
Whitmore, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3429.
Bourguel, *Bull. soc. chim.*, 1927, **41**, 1476.
Geldof, Wibaut, *Rec. trav. chim.*, 1948, **67**, 105.

2-Octene (2-*Octylene*)

$CH_3\cdot[CH_2]_4\cdot CH{:}CH\cdot CH_3$

C_8H_{16} MW, 112

Cis:
F.p. — 104°. B.p. 124·6°/750 mm. D_4^{20} 0·72287. n_D^{20} 1·41391.

Trans:
F.p. — 88°. B.p. 123·35°/750 mm. D_4^{20} 0·71842. n_D^{20} 1·41281.

Whitmore, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3430.
Klepper, *Chimie et Industrie*, 1929, Special No., 261.
Maman, *Chem. Zentr.*, 1936, I, 2332.
Bourguel, *Bull. soc. chim.*, 1927, **41**, 1476.
Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 2683.

3-Octene (3-*Octylene*)

$CH_3\cdot[CH_2]_3\cdot CH{:}CH\cdot CH_2\cdot CH_3$

C_8H_{16} MW, 112

Cis:
F.p. — 126°. B.p. 122·3°/741 mm. D_4^{20} 0·71888. n_D^{20} 1·41246.

Trans:
F.p. — 108°. B.p. 122·4°/741 mm. D_4^{20} 0·71630. n_D^{20} 1·41241.

Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 216, 2684.
Jacobs, *Organic Reactions*, 1949, **5**, 60 (*Review*).

4-Octene (4-*Octylene*)

$CH_3\cdot[CH_2]_2\cdot CH{:}CH\cdot[CH_2]_2\cdot CH_3$

C_8H_{16} MW, 112

Cis:
F.p. — 118°. B.p. 121·7°/739 mm. D_4^{20} 0·72048. n_D^{20} 1·41361.

Trans:
F.p. — 94°. B.p. 121·4°/739 mm. D_4^{20} 0·71467. n_D^{20} 1·41157.

Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 216, 2684.

Octene-carboxylic Acid.
See Nonenic Acid.

4-Octene-2 : 7-dione (2 : 7-*Diketo*-4-*octene*, 1 : 4-*diaceto*-2-*butylene*)

$CH_3\cdot CO\cdot CH_2\cdot CH{:}CH\cdot CH_2\cdot CO\cdot CH_3$

$C_8H_{12}O_2$ MW, 140

Cryst. M.p. 30–1°. B.p. 70–5°/0·03 mm. Sol. Et_2O. Insol. pet. ether. Reduces $NH_3.AgNO_3$.

Karrer, Eugster, *Helv. Chim. Acta*, 1949, **32**, 1935.

1-Octenic Acid (2-n-*Amylacrylic acid*, 1-*heptene*-1-*carboxylic acid*)

$CH_3\cdot[CH_2]_4\cdot CH{:}CH\cdot COOH$

$C_8H_{14}O_2$ MW, 142

Nitrile: $C_8H_{13}N$. MW, 123. (*a*) *Cis*: b.p. 74–5°/11 mm. D_4^{20} 0·82854. n_D^{20} 1·44032. (*b*) *Trans*: m.p. 57·6–58°. B.p. 84°/11 mm. D_4^{20} 0·82815. n_D^{20} 1·44344.

Amide: $C_8H_{15}ON$. MW, 141. (*a*) *Cis*: m.p. 66·0–66·4°. (*b*) *Trans*: m.p. 134·7–135·1°.

Bruylants, Fonteyn, *Chem. Abstracts*, 1933, **27**, 5717.
Delaby, Hubert, *Bull. soc. chim.*, 1943, **10**, 576.

2-Octenic Acid (2-*Heptene*-1-*carboxylic acid*)

$CH_3\cdot[CH_2]_3\cdot CH{:}CH\cdot CH_2\cdot COOH$

$C_8H_{14}O_2$ MW, 142

Nitrile: (*trans*), b.p. 93–5°/20 mm. D_0 0·855. n_D^{23} 1·4369.

Delaby, Lecomte, *Bull. soc. chim.*, 1937, **4**, 749.
Delaby, Hubert, *Bull. soc. chim.*, 1943, **10**, 573, 576.

6-Octenic Acid (1-*Heptene*-7-*carboxylic acid*)

$CH_2{:}CH\cdot[CH_2]_4\cdot CH_2\cdot COOH$

$C_8H_{14}O_2$ MW, 142

Me ester: $C_9H_{16}O_2$. MW, 156. B.p. 190°/760 mm.
Et ester: $C_{10}H_{18}O_2$. MW, 170. B.p. 210°.
p-*Bromophenacyl ester*: m.p. 58°.

Carmichael, *J. Chem. Soc.*, 1922, **121**, 2549.
Baudert, *Bull. soc. chim.*, 1946, **13**, 85.

1-Octenol-2 (2-*Hydroxy*-1-*octene*)

$CH_3\cdot[CH_2]_5\cdot C(OH){:}CH_2$

$C_8H_{16}O$ MW, 128

Acetyl: b.p. 108°/42·5 mm. D_{20}^{25} 0·8692. n_D^{20} 1·4283.

Me ether: $C_9H_{18}O$. MW, 142. B.p. 166–8°. D_4^{16} 0·8170. n_D^{16} 1·4309.

Moureu, *Compt. rend.*, 1904, **138**, 287.
Gwynn, Degering, U.S.P. 2,386,058, (*Chem. Abstracts*, 1946, **40**, 346).

1-Octenol-3 (3-*Hydroxy*-1-*octene, amylvinylcarbinol*)

$CH_3{\cdot}[CH_2]_4{\cdot}CH(OH){\cdot}CH{:}CH_2$

$C_8H_{16}O$ MW, 128

B.p. 173·5°/742 mm., 68–71°/12 mm., 55–55·5°/5 mm. D_4^{13} 0·8395. n_D^{12} 1·4391. Occurs in fungus *matsutake*.

d-.
$[\alpha]_D^{17}$ +10·7° in EtOH, $[\alpha]_D^{17}$ + 5·7° in $CHCl_3$.

l-.
$[\alpha]_D^{17}$ − 13·1° in EtOH, $[\alpha]_D^{17}$ − 6·7° in $CHCl_3$, $[\alpha]_D^{15}$ − 17°. Acetylation does not cause racemisation.

Acetyl: b.p. 190°/746 mm., 65–6°/5·5 mm. D_4^{20} 0·873. n_D^{20} 1·4223. $[\alpha]_D^{20}$ + 3·5°. Occurs in essence of lavender.

Acid phthalate: m.p. 57° (from natural alcohol), m.p. 79° (from synthetic alcohol).

4′-*Iododiphenylurethane*: m.p. 165·5–166°.

Murahashi, *Chem. Abstracts*, 1938, **32**, 3755.
Crabalona, *Bull. soc. chim.*, 1944, **11**, 67.

1-Octenol-4 (4-*Hydroxy*-1-*octene, butylallylcarbinol*)

$CH_3{\cdot}[CH_2]_3{\cdot}CH(OH){\cdot}CH_2{\cdot}CH{:}CH_2$

$C_8H_{16}O$ MW, 128

B.p. 171·5–172° (corr.)/748 mm., 68–9°/10 mm. D_4^{20} 0·8373. n_D^{20} 1·4383.

Henze, Allen, Leslie, *J. Org. Chem.*, 1942, **7**, 329.

2-Octenol-4 (4-*Hydroxy*-2-*octene, butylpropenylcarbinol*)

$CH_3{\cdot}[CH_2]_3{\cdot}CH(OH){\cdot}CH{:}CH{\cdot}CH_3$

$C_8H_{16}O$ MW, 128

B.p. 175°/760 mm., 93·9–95·9°/40 mm., 79–80°/23 mm., 83°/20 mm., 74°/8 mm. D_4^{20} 0·8393 (0·8427). n_D^{20} 1·4395 (1·4420), n_D^{15} 1·4435.

3 : 5-*Dinitrobenzoyl*: plates from EtOH.Aq. M.p. 54°.

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, **57**, 1606.
Hargreaves, Owen, *J. Chem. Soc.*, 1947, 757.

2-Octenol-8 (8-*Hydroxy*-2-*octene*)

$HO{\cdot}CH_2{\cdot}[CH_2]_4{\cdot}CH{:}CH{\cdot}CH_3$

$C_8H_{16}O$ MW, 128

B.p. 187–93°. Sol. EtOH, Et_2O. Spar. sol. H_2O.

Löbl, *Monatsh.*, 1903, **24**, 398.

3-Octenol-1 (1-*Hydroxy*-3-*octene*)

$CH_3{\cdot}[CH_2]_3{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CH_2OH$

$C_8H_{16}O$ MW, 128

B.p. 86°/12 mm. D_{16}^{12} 0·849.

Acetyl: b.p. 93°/12 mm. D_{10}^{13} 0·893.

Phenylurethane: m.p. 30°. B.p. 204°/12 mm.

Normant, *Compt. rend.*, 1948, **226**, 733.

4-Octenol-1 (1-*Hydroxy*-4-*octene*)

$CH_3{\cdot}[CH_2]_2{\cdot}CH{:}CH{\cdot}[CH_2]_2{\cdot}CH_2OH$

$C_8H_{16}O$ MW, 128

B.p. 95°/17 mm. D_4^{20} 0·842. n_D^{20} 1·4462 Dist. with conc. H_2SO_4 ⟶ 2-butyltetrahydrofuran.

Acetyl: b.p. 101°/19 mm. D_4^{14} 0·89. n_D^{14} 1·4356.

3 : 5-*Dinitrobenzoyl*: m.p. 44°.

Allophanate: b.p. 160°.

Riobé, *Compt. rend.*, 1947, **225**, 334.

2-Octenone-4 (4-*Keto*-2-*octene, butyl propenyl ketone*)

$CH_3{\cdot}[CH_2]_3{\cdot}CO{\cdot}CH{:}CH{\cdot}CH_3$

$C_8H_{14}O$ MW, 126

B.p. 178°/740 mm. D_4^{20} 0·852. n_D^{25} 1·4393.

2 : 4-*Dinitrophenylhydrazone*: orange needles from 95% EtOH. M.p. 108–9°.

Stutsman, Adkins, *J. Am. Chem. Soc.*, 1939, **61**, 3306.

3-Octenone-5 (*Propylidenemethyl* n-*propyl ketone*, 5-*keto*-3-*octene*)

$CH_3{\cdot}[CH_2]_2{\cdot}CO{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CH_3$

$C_8H_{14}O$ MW, 126

B.p. 68–72°/25 mm.

Benary, *Ber.*, 1931, **64**, 2544.

Octinum (*Octin*, 6-*methylamino*-2-*methylheptene*-2)

$(CH_3)_2C{:}CH{\cdot}[CH_2]_2{\cdot}CH(CH_3){\cdot}NH{\cdot}CH_3$

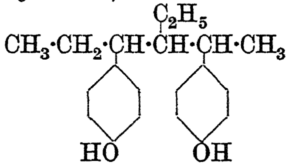
$C_9H_{19}N$ MW, 141

B.p. 176–8°. Soporific.

Picrate: m.p. 70°.

Knoll, Danish P., 48,717, (*Chem. Zentr.*, 1934, II, 1493).

Octofollin (*Benzestrol*, 2 : 4-*di*-(p-*hydroxyphenyl*)-3-*ethylhexane*)



$C_{20}H_{26}O_2$ MW, 298

Colourless cryst. M.p. 162°. Possesses high œstrogenic activity.

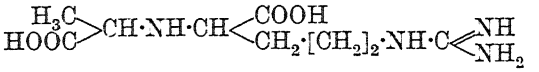
Di-Me ether: m.p. 56°.

Stuart, Shukis, Tallman, McCann, Treves, *J. Am. Chem. Soc.*, 1946, **68**, 729.

n-Octoic Acid.

See n-Caprylic Acid.

Octopin (*Pectenin*, l-*arginine*-l-α-*propionic acid*)



$C_9H_{18}O_4N_4$ MW, 246

Isolated from aq. extracts of tentacle muscles of *Loligo pealii* and *Octopus vulgaris*, the adductor muscles of the scallop *Pecten magellanicus* and the muscles of the octopod *Eledone moschata*. Needles from H_2O or EtOH.Aq. M.p. 281–2° (261–4°). Odourless and tasteless. Readily sol. hot H_2O. Spar. sol. MeOH, EtOH, Et_2O. $[\alpha]_D^{17}$ + 20·94° in H_2O.

Cu salt: m.p. 223–7° decomp.

Ni salt: m.p. above 290°.

Picrate: m.p. 226–30° corr. decomp.

Picrolonate: m.p. 237–9° corr. decomp.

Akashi, *Chem. Abstracts*, 1937, **31**, 5766.
Knoop, Martius, *Z. physiol. Chem.*, 1938, **254**, 1; 1939, **258**, 238.
Irvin, Wilson, *J. Biol. Chem.*, 1939, **127**, 555, 565, 575.
Karrer, Koenig, Legler, *Helv. Chim. Acta*, 1941, **24**, 127.
Herbst, Swart, *J. Org. Chem.*, 1946, **11**, 368.

Octopine.

See Carnegine.

n-Octyl Alcohol (1-*Octanol*, 1-*hydroxy*-n-*octane*)

$CH_3 \cdot [CH_2]_6 \cdot CH_2OH$

$C_8H_{18}O$ MW, 130

M.p. − 16·7°. B.p. 194·45° (195–8°, 195–7°), 98°/19 mm., 90·2°/11·8 mm., 82·8°/7·5 mm. D_4^{20} 0·8270. $n_D^{20.5}$ 1·43035. Heat of comb. C_p 1265·0 Cal., C_v 1262·7 Cal.

p-*Nitrobenzoyl*: m.p. 166°. B.p. 170–5°/5 mm. D_{20}^{20} 1·0719. n_D^{20} 1·5074.

3 : 5-*Dinitrobenzoyl*: m.p. 61–2°.

Me ether: *see* Methyl octyl Ether.

Et ether: $C_{10}H_{22}O$. MW, 158. B.p. 189·2°, 72–3°/8 mm.

n-*Propyl ether*: $C_{11}H_{24}O$. MW, 172. B.p. 207°.

n-*Butyl ether*: $C_{12}H_{26}O$. MW, 186. B.p. 225–7°.

n-*Heptyl ether*: $C_{15}H_{32}O$. MW, 228. B.p. 278·8°.

Phenylurethane: m.p. 74–74·5° (72°).

o-*Nitrophenylurethane*: m.p. 44°.

m-*Nitrophenylurethane*: m.p. 63°.

3 : 5-*Dinitrophenylurethane*: m.p. 69°.

Blaise, Picard, *Compt. rend.*, 1911, **152**, 269.
Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, **15**, 267.
Schrauth, Schenck, Stickdorn, *Ber.*, 1931, **64**, 1318.
Goebel, Marvel, *J. Am. Chem. Soc.*, 1933, **55**, 1696.
Vaughn, Spahr, Nieuwland, *J. Am. Chem. Soc.*, 1933, **55**, 4207.
Deutsche Gold- u. Silber-Scheideanstalt, B.P. 381,185, (*Chem. Abstracts*, 1933, **27**, 3943).
Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, **55**, 3825.
Deffert, *Bull. soc. chim. Belg.*, 1931, **40**, 385.
Ueno, Komori, *Chem. Abstracts*, 1948, **42**, 6738.

sec.-n-Octyl Alcohol (*Methyl*-n-*hexylcarbinol*, 2-*hydroxy*-n-*octane*, *octanol*-2, *capryl alcohol*)

$CH_3 \cdot [CH_2]_5 \cdot CH(OH) \cdot CH_3$

$C_8H_{18}O$ MW, 130

d-.

B.p. 86°/20 mm. $[\alpha]_D^{17}$ + 9·9°. D_4^{20} 0·8216.

l-.

B.p. 86°/20 mm. $[\alpha]_D^{17}$ − 9·9°.

dl-.

B.p. 179·5°, 110°/120 mm. $D_4^{25.2}$ 0·8188. n_D^{20} 1·42025.

Formyl: $C_9H_{18}O_2$. MW, 158. B.p. 184°/744 mm. D_4^{14} 0·8642.

Acetyl: $C_{10}H_{20}O_2$. MW, 172. B.p. 194·5°/744 mm., 84°/15 mm. D_4^{19} 0·8606. n_D^{20} 1·4141.

Bromoacetyl: $C_{10}H_{19}O_2Br$. MW, 251. B.p. 137°/18 mm.

Propionyl: $C_{11}H_{22}O_2$. MW, 186. B.p. 211°/744 mm., 96°/15 mm. $D_{44}^{20.5}$ 0·8650. n_D^{20} 1·4168.

Butyryl: $C_{12}H_{24}O_2$. MW, 200. B.p. 220°/744 mm., 115°/18 mm. D_4^{17} 0·8587. n_D^{20} 1·4202.

Isobutyryl: b.p. 220°/744 mm. D_4^{14} 0·8554.

Valeryl: $C_{13}H_{26}O_2$. MW, 214. B.p. 127°/17 mm. $D_4^{16.5}$ 0·8560. n_D^{20} 1·4225.

Isovaleryl: b.p. 236·5°/744 mm. D_4^{14} 0·8540.

d-*Caproyl*: $C_{14}H_{28}O_2$. MW, 228. B.p. 141°/17 mm. D_4^{12} 0·8598. n_D^{20} 1·4264.

d-*Heptylyl*: $C_{15}H_{30}O_2$. MW, 242. B.p. 151°/15 mm. D_4^{13} 0·8593. n_D^{20} 1·4290.

d-*Caprylyl*: $C_{16}H_{32}O_2$. MW, 256. B.p. 165°/18 mm. D_4^{12} 0·8557. n_D^{20} 1·4308.

d-*Nonoyl*: $C_{17}H_{34}O_2$. MW, 270. B.p. 174°/17 mm. D_4^{22} 0·8542. n_D^{20} 1·4342.

d-*Undecylyl*: $C_{19}H_{38}O_2$. MW, 298. B.p. 167°/3 mm. D_4^{19} 0·8538. n_D^{20} 1·4373.

d-*Lauryl*: $C_{20}H_{40}O_2$. MW, 312. B.p. 183°/7 mm. $D_4^{16.5}$ 0·8585. n_D^{20} 1·4393.

d-*Myristyl*: $C_{22}H_{44}O_2$. MW, 340. B.p. 197°/4 mm. D_4^{17} 0·8562. n_D^{20} 1·4416.

d-*Palmityl*: $C_{24}H_{48}O_2$. MW, 368. M.p.

32°. B.p. 216°/18 mm. D_4^{45} 0·8372. n_D^{20} 1·4438.

d-*Stearyl*: $C_{26}H_{52}O_2$. MW, 396. M.p. 34°. B.p. 235°/6 mm. D_4^{38} 0·8410.

Phenylacetyl: $C_{16}H_{24}O_2$. MW, 248. B.p. 195°/35 mm., 174°/12 mm. D_4^{20} 0·9578.

Benzoyl: $C_{15}H_{22}O_2$. MW, 234. B.p. 175°/2–5 mm. D_4^{20} 1·0185. n_D^{20} 1·5476.

l-o-*Aminobenzoyl*: $C_{15}H_{23}O_2N$. MW, 249. B.p. 183°/10 mm.

l-m-*Aminobenzoyl*: b.p. 195–9°/18 mm.

l-p-*Aminobenzoyl*: m.p. 69–70°. B.p. 200°/10 mm. *B,HCl*: m.p. 131–3°.

Salicyloyl: $C_{15}H_{22}O_3$. MW, 250. B.p. 170°/10 mm.

Me ether: $C_9H_{20}O$. MW, 144. B.p. 76–7°/44 mm. D_4^{20} 0·8094. n_D^{25} 1·4212

Et ether: $C_{10}H_{22}O$. MW, 158. B.p. 63–5°/14 mm. D_4^{20} 0·7861. n_D^{25} 1·4136.

n-*Propyl ether*: $C_{11}H_{24}O$. MW, 172. B.p. 76°/18 mm. D_4^{20} 0·7887. n_D^{25} 1·4148.

n-*Butyl ether*: $C_{12}H_{26}O$. MW, 186. B.p. 85–6°/14 mm. D_4^{20} 0·7923. n_D^{25} 1·4168.

n-*Amyl ether*: $C_{13}H_{28}O$. MW, 200. B.p. 99°/15 mm. D_4^{20} 0·7958. n_D^{25} 1·4218.

n-*Hexyl ether*: $C_{14}H_{30}O$. MW, 214. B.p. 115°/15 mm. D_4^{20} 0·7983. n_D^{25} 1·4252.

n-*Heptyl ether*: $C_{15}H_{32}O$. MW, 228. B.p. 129°/18 mm. D_4^{20} 0·8017. n_D^{25} 1·4267.

n-*Octyl ether*: $C_{16}H_{34}O$. MW, 242. B.p. 146°/13 mm. D_4^{20} 0·8038. n_D^{25} 1·4301.

n-*Nonyl ether*: $C_{17}H_{36}O$. MW, 256. B.p. 163°/18 mm. D_4^{20} 0·8042. n_D^{25} 1·4325.

Benzyl ether: $C_{15}H_{24}O$. MW, 220. B.p. 154°/18 mm. D_4^{20} 0·8974.

Diphenylmethyl ether: $C_{21}H_{28}O$. MW, 296. B.p. 202°/10 mm. D_4^{17} 0·9675.

Triphenylmethyl ether: $C_{27}H_{32}O$. MW, 372. B.p. 178°/0·3 mm. D_4^{16} 1·026.

Kenyon, McNicol, *J. Chem. Soc.*, 1923, **123**, 18.
Kenyon, *Organic Syntheses*, 1926, VI, 68.
Waterman, te Nuyl, *Rec. trav. chim.*, 1932, **51**, 533.
Ellis, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 1678.

n-Octylamine (1-*Amino*-n-*octane*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2NH_2$

$C_8H_{19}N$ MW, 129

B.p. 175–7°/745 mm. (179–80°, 185–7°). $D^{20\cdot8}$ 0·7769. $k = 4{\cdot}5 \times 10^{-4}$ in H_2O at 25°.

Picrate: m.p. 111·5–112·5°.

Nyssens, *Chem. Abstracts*, 1931, **25**, 70.
Adamson, Kenner, *J. Chem. Soc.*, 1934, 842.

***sec.-n*-Octylamine.**

See 2-Amino-*n*-octane.

***n*-Octylbenzene** (1-*Phenyl*-n-*octane*)

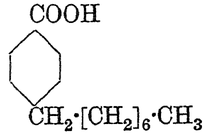
$C_6H_5{\cdot}[CH_2]_7{\cdot}CH_3$

$C_{14}H_{22}$ MW, 190

F.p. − 7°. B.p. 264–5° (262–4°), 131–4°/12 mm. D_4^{20} 0·8582, D_4^{30} 0·8512. n_D^{20} 1·4851.

Sabatier, Maihle, *Compt. rend.*, 1914, **158**, 834.
Tilicheev, Kuruindin, *Chem. Abstracts*, 1931, **25**, 3469.
Eisenlohr, Schultz, *Ber.*, 1924, **57**, 1815.
Schmidt, Hopp, Schoeller, *Ber.*, 1939, **72**, 1893.

***p*-Octylbenzoic Acid**



$C_{15}H_{22}O_2$ MW, 234

Leaflets from hot EtOH. M.p. 139°. (White cryst. from EtOH.Aq. M.p. 99–100°.) Spar. sol. H_2O.

Beran, *Ber.*, 1885, **18**, 138.
Zaki, Fahim, *J. Chem. Soc.*, 1942, 308.

***n*-Octyl bromide** (1-*Bromo*-n-*octane*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2Br$

$C_8H_{17}Br$ MW, 193

M.p. − 55·0°. B.p. 201·5°. D_4^{25} 1·10788. n_D^{25} 1·4503. Characterised by conversion into *n*-octylthiouronium picrate, yellow cryst. from EtOH. M.p. 134°.

Vogel, *J. Chem. Soc.*, 1943, 644.
Kubiczek, Pohl, Smahel, *Monatsh.*, 1947, **77**, 52.
Kamm, Marvel, *Organic Syntheses*, 1921, I, 7; Collective Vol. I, 28.

***sec.-n*-Octyl bromide** (2-*Bromo*-n-*Octane*)

$CH_3{\cdot}[CH_2]_5{\cdot}CHBr{\cdot}CH_3$

$C_8H_{17}Br$ MW, 193

d-.

B.p. 71°/14 mm., 60°/3 mm. D_4^{25} 1·0982. n_D^{25} 1·4475. $[\alpha]_D^{25}$ + 31·0° (no solvent), (+ 34·2°, + 29·8°).

l-.

B.p. 83–4°/18 mm. D_4^{20} 1·092. n_D^{20} 1·4500. $[\alpha]_D^{25}$ − 30·5° (no solvent), (− 40·6°, − 34·25°, − 33·1°).

dl-.

B.p. 72°/14 mm., 61°/3 mm. D_4^{25} 1·0878 (1·09681). n_D^{25} 1·4442 (1·4482).

Ellis, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 1680.
Rule, Smith, Harrower, *J. Chem. Soc.*, 1933, 386.
Kornblum *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 309.
Reynolds, Adkins, *J. Am. Chem. Soc.*, 1929, **51**, 279.
Tseng, Hau, Hu, *Chem. Zentr.*, 1936, I, 2917.
Gerrard, *J. Chem. Soc.*, 1946, 744.

n-Octyl chloride (1-*Chloro*-n-*octane*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2Cl$

$C_8H_{17}Cl$ MW, 148·5

B.p. 181·5°/765 mm., 78°/15 mm. D_4^{20} 0·8748, $D_4^{56\cdot6}$ 0·8192, D_0^4 0·892. n_D^{20} 1·43058.

Bouveault, Blanc, *Bull. soc. chim.*, 1904, **31**, 673.
Clark, Streight, *Chem. Abstracts*, 1930, **24**, 586.
Vogel, *J. Chem. Soc.*, 1943, 640.

sec.-n-Octyl chloride (2-*Chloro*-n-*octane*)

$CH_3{\cdot}[CH_2]_5{\cdot}CHCl{\cdot}CH_3$

$C_8H_{17}Cl$ MW, 148·5

d-.
B.p. 75°/28 mm., 55–6°/10–11 mm. D_4^{17} 0·8658. n_D^{21} 1·4273. $[\alpha]_D^{17}$ + 20·40° ($[\alpha]_D^{20}$ + 33·70°).

l-.
B.p. 70°/25 mm., 68°/21 mm., 60°/12 mm. D_4^{17} 0·8628. n_D^{20} 1·4302. $[\alpha]_D^{16}$ − 29·8° (− 38·2°), $[\alpha]_D^{20}$ − 36·15°.

dl-.
B.p. 171–3°. D^{15} 0·87075.

Houssa, Phillips, *J. Chem. Soc.*, 1932, 109.
Malbot, *Bull. soc. chim.*, 1890, **3**, 69.
Houssa, Kenyon, Phillips, *J. Chem. Soc.*, 1929, 1700.
Rule, Smith, Harrower, *J. Chem. Soc.*, 1933, 386.
Gerrard, *J. Chem. Soc.*, 1945, 109.

Octylene.
See Octene.

sec.-n-Octyl fluoride (2-*Fluoro*-n-*octane*)

$CH_3{\cdot}[CH_2]_5{\cdot}CHF{\cdot}CH_3$

$C_8H_{17}F$ MW, 132

Unstable liq. Cannot be distilled without decomp.

Simons, Bassler, *J. Am. Chem. Soc.*, 1941, **63**, 880.

Octyl hexadecyl Ketone (*Pentacosanone*-9)

$CH_3{\cdot}[CH_2]_{15}{\cdot}CO{\cdot}[CH_2]_7{\cdot}CH_3$

$C_{25}H_{50}O$ MW, 366

Powder from C_6H_6. M.p. 66°. Sol. EtOH.
Semicarbazone: m.p. 39–41°.

Brigl, *Z. physiol. Chem.*, 1915, **95**, 178.

n-Octyl iodide (1-*Iodo*-n-*octane*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2I$

$C_8H_{17}I$ MW, 240

M.p. − 45·7°. B.p. 225–225·5°, 194°/330 mm., 86·5°/5 mm. D_4^{15} 1·33568. n_D^{20} 1·48892.

Vogel, *J. Chem. Soc.*, 1943, 647.

sec.-n-Octyl iodide (2-*Iodo*-n-*octane*)

$CH_3{\cdot}[CH_2]_5{\cdot}CHI{\cdot}CH_3$

$C_8H_{17}I$ MW, 240

d-.
B.p. 101°/22 mm. D_4^{17} 1·3314. $[\alpha]_D^{17}$ + 39·83°.

l-.
B.p. 92°/12 mm. D_4^{17} 1·3299. $[\alpha]_D^{17}$ − 40·56°, $[\alpha]_D^{20}$ − 47·87°.

dl-.
B.p. 190° (210°) part. decomp. $D_{15}^{17\cdot5}$ 1·318.

Pickard, Kenyon, *J. Chem. Soc.*, 1911, **99**, 69.
Cf. Houssa, Kenyon, Phillips, *J. Chem. Soc.*, 1929, 1700.

n-Octylmalonic Acid

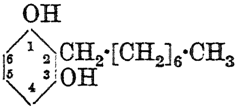
$CH_3{\cdot}[CH_2]_7{\cdot}CH{<}^{COOH}_{COOH}$

$C_{11}H_{20}O_4$ MW, 216

Prisms from C_6H_6–pet. ether. M.p. 116° (113–14°).
Di-Et ester: $C_{15}H_{28}O_4$. MW, 272. B.p. 175–7°/17 mm., 150–3°/7 mm. n_D^{25} 1·4315.

Robinson, *J. Chem. Soc.*, 1924, **125**, 228.
Clutterbuck, Raistrick, Rintoul, *Trans. Roy. Soc.*, 1931, **B220**, 301.
Floyd, Miller, *J. Am. Chem. Soc.*, 1947, **69**, 2355.

2-n-Octylresorcinol (2 : 6-*Dihydroxyoctylbenzene*)



$C_{14}H_{22}O_2$ MW, 222

White needles from CCl_4 or C_6H_6–ligroin. M.p. 55–6°. Sol. C_6H_6, EtOH, hot CCl_4. Insol. ligroin. No col. with $FeCl_3$.

Russell, Gulledge, *J. Am. Chem. Soc.*, 1942, **64**, 1314.

4-n-Octylresorcinol (2 : 4-*Dihydroxyoctylbenzene*).

M.p. 74–5°. B.p. 199–201°/6–7 mm.

Dohme, Cox, Miller, *J. Am. Chem. Soc.*, 1926, **48**, 1692.

5-n-Octylresorcinol (3 : 5-*Dihydroxyoctylbenzene*).

Colourless cryst. from ligroin. M.p. 61–2° corr. B.p. 195–7°/2 mm.
Di-Me ether: colourless oil. B.p. 164–8°/4 mm. n_D^{20} 1·4995.

Adams, Loewe, Jelinek, Wolff, *J. Am. Chem. Soc.*, 1941, **63**, 1972.

n-Octyl thiocyanate

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2{\cdot}CNS$

$C_9H_{17}NS$ MW, 171

B.p. 141–2°/19 mm. D_4^{25} 0·9149. n_D^{25} 1·4642.

Allen, *J. Am. Chem. Soc.*, 1935, **57**, 198.

1-Octyl-2-undecyl-ethylene.
See 9-Heneicosene.

1-Octyne (n-*Hexylacetylene*)

$CH_3\cdot[CH_2]_5\cdot C⫶CH$

C_8H_{14} MW, 110

F.p. − 79·48° (− 79°). B.p. 126·25°/760 mm., 125·2°/737 mm. D_4^{20} 0·7460 (0·74629). n_D^{20} 1·4159 (1·41565).

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, **57**, 1607.
Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 485.
Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 2684.

2-Octyne (*Methyl*-n-*amylacetylene*)

$CH_3\cdot[CH_2]_4\cdot C⫶C\cdot CH_3$

C_8H_{14} MW, 110

F.p. − 61·6° (− 62°). B.p. 138·00°/760 mm., 137·1°/742 mm. D_4^{20} 0·7596 (0·75913). n_D^{20} 1·4278 (1·42697).

Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 485.
Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 2684.
Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, **57**, 1607.

3-Octyne (*Ethyl*-n-*butylacetylene*)

$CH_3\cdot[CH_2]_3\cdot C⫶C\cdot CH_2\cdot CH_3$

C_8H_{14} MW, 110

F.p. − 103·9° (− 105°). B.p. 133·14°/760 mm., 132·8–132·9°/747 mm. D_4^{20} 0·7522 (0·75291). n_D^{20} 1·4250.

Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 485.
Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 2684.

4-Octyne (*Di*-n-*propylacetylene*)

$CH_3\cdot[CH_2]_2\cdot C⫶C\cdot[CH_2]_2\cdot CH_3$

C_8H_{14} MW, 110

F.p. − 102·55° (− 102°). B.p. 131·57°/760 mm. D_4^{20} 0·7509 (0·75124). n_D^{20} 1·4248 (1·42431).

Johnson, *J. Chem. Soc.*, 1946, 1013.
Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 485.
Campbell, Eby, *J. Am. Chem. Soc.*, 1941, **63**, 2684.

2-Octynol-1 (*Amylpropargyl alcohol*, 1-*hydroxy*-2-*octyne*)

$CH_3\cdot[CH_2]_4\cdot C⫶C\cdot CH_2OH$

$C_8H_{14}O$ MW, 126

Liq. B.p. 98–9°/15 mm. D_4^{17} 0·884. n_D^{17} 1·4585.

Acetyl: liq. B.p. 79–81°/6 mm. D_4^{20} 0·9261. n_D^{20} 1·4440.

Moureu, Desmots, *Compt. rend.*, 1901, **132**, 1224.
Lai, *Bull. soc. chim.*, 1933, **53**, 682.
Hennion, Bell, *J. Am. Chem. Soc.*, 1943, **65**, 1847.

3-Octynol-2 (2-*Hydroxy*-3-*octyne*, *amyl*-1-*hydroxyethylacetylene*)

$CH_3\cdot[CH_2]_3\cdot C⫶C\cdot CH(OH)\cdot CH_3$

$C_8H_{14}O$ MW, 126

Liq. B.p. 101·5–103°/40 mm. $n_D^{17.5}$ 1·4503.

1-*Naphthylurethane*: needles from ligroin. M.p. 63·5–64·5°.

Campbell, Campbell, Eby, *J. Am. Chem. Soc.*, 1938, **60**, 2882.
Bowden, Heilbron, Jones, Weedon, *J. Chem. Soc.*, 1946, 45.

3-Octynone-2 (2-*Keto*-3-*octyne*, 1-*aceto*-1-*hexyne*)

$CH_3\cdot[CH_2]_3\cdot C⫶C\cdot CO\cdot CH_3$

$C_8H_{12}O$ MW, 124

Liq. B.p. 74°/12 mm. n_D^{16} 1·4487.

Semicarbazone: needles from EtOH.Aq. M.p. 109°.

Phenylsemicarbazone: needles from EtOH. Aq. M.p. 104–104·5°.

2 : 4-*Dinitrophenylhydrazone*: needles from EtOH.Aq. M.p. 87–8°.

Kroeger, Nieuwland, *J. Am. Chem. Soc.*, 1936, **58**, 1861.
Jones *et al.*, *J. Chem. Soc.*, 1946, 43.

Œnanthaldehyde.
See n-Heptaldehyde.

Œnanthetol

$HO\cdot CH_2\cdot CH⁚CH\cdot[C⫶C]_2\cdot[CH⁚CH]_2\cdot[CH_2]_5\cdot CH_3$

or

$HO\cdot CH_2\cdot[CH⁚CH]_2\cdot[C⫶C]_2\cdot CH⁚CH\cdot[CH_2]_5\cdot CH_3$

$C_{17}H_{22}O$ MW, 242

Occurs in *Œnanthe crocata*. Cryst. M.p. 71°. $[\alpha]_D$ 0°.

Anet, Lythgoe, Silk, Trippett, *Chemistry and Industry*, 1952, 757.

Œnanthetone

$CH_3\cdot CH⁚CH\cdot[C⫶C]_2\cdot[CH⁚CH]_2\cdot CH_2\cdot CH_2\cdot CO$
$\qquad\qquad\qquad\qquad\qquad\qquad CH_3.CH_2\cdot CH_2$

or

$CH_3\cdot[CH⁚CH]_2\cdot[C⫶C]_2\cdot CH⁚CH\cdot CH_2\cdot CH_2\cdot CO$
$\qquad\qquad\qquad\qquad\qquad\qquad CH_3\cdot CH_2\cdot CH_2$

$C_{17}H_{20}O$ MW, 240

Occurs in *Œnanthe crocata*. Cryst. M.p. 46°.

Anet, Lythgoe, Silk, Trippett, *Chemistry and Industry*, 1952, 757.

Œnanthic Acid.
See n-Heptylic Acid.

Œnanthol.
See n-Heptaldehyde.

Œnanthonaphthone.
See Hexyl 2-naphthyl Ketone.

Œnanthone.
See Di-*n*-hexyl Ketone.

Œnanthophenone.
See Hexyl phenyl Ketone.

Œnanthotoxin

$HO{\cdot}CH_2{\cdot}CH{:}CH{\cdot}[C{:}C]_2{\cdot}[CH{:}CH]_2{\cdot}CH_2{\cdot}CH_2$
$CH_3{\cdot}[CH_2]_2{\cdot}CH(OH)$
or
$HO{\cdot}CH_2{\cdot}[CH{:}CH]_2{\cdot}[C{:}C]_2{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CH_2$
$CH_3{\cdot}[CH_2]_2{\cdot}CH(OH)$

$C_{17}H_{22}O_2$ MW, 258

Convulsant poison occurring in *Œnanthe crocata*. Cryst. M.p. 87°. $[\alpha]_D$ +30·5° in MeOH. UV. maxima at 213, 252, 267, 281 (inflexion), 296, 315·5 and 337·5 mμ.

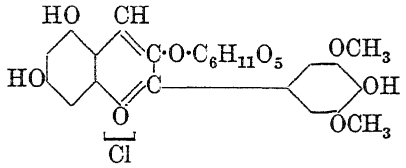
Jacobson, *J. Am. Chem. Soc.*, 1915, **37**, 916.
Clarke, Kidder, Robertson, *J. Pharm. Pharmacol.*, 1949, **1**, 377.
Anet, Lythgoe, Silk, Trippett, *Chemistry and Industry*, 1952, 757.

Œnanthylic Acid.
See *n*-Heptylic Acid.

Œnanthylidene.
See 1-Heptyne.

Œnidin chloride.
See Malvidin chloride.

Œnin chloride (*Primulin chloride, malvidin chloride* 3-β-*glucoside*)



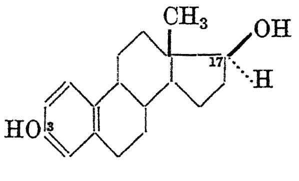
$C_{23}H_{25}O_{12}Cl$ MW, 528·5

Pigment of skins of black grapes. Violet cryst. by transmitted light with yellow metallic cast. Bronze appearance in masses. Gives bluish-violet smear on paper or porcelain.

Picrate: red needles with green reflex. Decomp. at 202°.

Kondo, *Chem. Abstracts*, 1930, **24**, 2748.
Levy, Posternack, Robinson, *J. Chem. Soc.*, 1931, 2701 (*Bibl.*).

"α"-**Œstradiol** (*Œstradiol*-17β, *estradiol*-17β)



$C_{18}H_{24}O_2$ MW, 272

Isolated from ovaries and pregnancy urine. Leaflets or needles from EtOH.Aq. Cryst. from Me_2CO. M.p. 178°. $[\alpha]_D^{18}$ +78° in EtOH. Digitonide is insol. 80% EtOH. The most potent of the natural œstrogens.

3-*Me ether*: needles from MeOH.Aq. M.p. 97–8°.

3-*Acetyl*: m.p. 137°.

17-*Acetyl*: m.p. 215–17°.

Diacetyl: leaflets from MeOH.Aq. M.p. 125–6°.

3-*Propionyl*: m.p. 125°.

17-*Propionyl*: m.p. 199–200°.

Dipropionyl: m.p. 104–5°.

17-n-*Butyryl*: m.p. 166–7°.

Di-n-butyryl: m.p. 64–5°.

3-*Trimethylacetyl*: needles from MeOH.Aq. M.p. 178–80°.

Di-trimethylacetyl: needles from Me_2CO–MeOH. M.p. 174–6°.

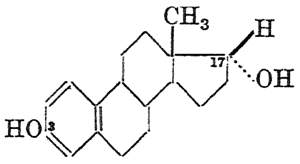
3-*Palmityl*: m.p. 69–71°.

3-*Benzoyl*: needles from EtOH.Aq. M.p. 192–3°.

3 : 17-*Dibenzoyl*: leaflets from EtOH. M.p. 168–9°.

Butenandt, *Z. physiol. Chem.*, 1937, **248**, 129.
Doisy, *J. Biol. Chem.*, 1936, **115**, 435.
Wintersteiner, *J. Biol. Chem.*, 1937, **118**, 789; *J. Am. Chem. Soc.*, 1937, **59**, 765.
Marker, *J. Am. Chem. Soc.*, 1938, **60**, 2927.
Goldberg, Sicé, Robert, Plattner, *Helv. Chim. Acta*, 1947, **30**, 1441.

"β"-**Œstradiol** (*Œstradiol*-17α, *estradiol*-17α)



$C_{18}H_{24}O_2$ MW, 272

Isolated from pregnancy urine. Needles from EtOH.Aq. Cryst. from Me_2CO. M.p. 223°. $[\alpha]_D^{18}$ +56·7° in EtOH. Much less potent than the "α"-isomer.

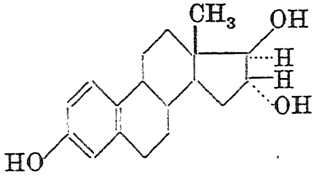
3-*Me ether*: leaflets from MeOH. M.p. 109–10°.

3 : 17-*Diacetyl*: needles from EtOH.Aq. M.p. 139–40°.

3-*Benzoyl*: leaflets from MeOH.Aq. M.p. 156–7°.

Butenandt, *Z. physiol. Chem.*, 1937, **248**, 129.
Wintersteiner, *J. Biol. Chem.*, 1938, **122**, 303; *J. Am. Chem. Soc.*, 1937, **59**, 765.
Marker, *J. Am. Chem. Soc.*, 1938, **60**, 2927.
Goldberg, Sicé, Robert, Plattner, *Helv. Chim. Acta*, 1947, **30**, 1441.

Œstriol (*Theelol, folliculin, follicular hormone hydrate*)



$C_{18}H_{24}O_3$ MW, 288

Isolated from urine of pregnancy. Cryst. from EtOH–AcOEt. M.p. 283° (275°). Sol. 33,000 parts H_2O. Sol. EtOH, Me_2CO, Py. Mod. sol. Et_2O. Spar. sol. pet. ether. Insol. alk. carbonates. Very sol. aq. alkalis. $[\alpha]_{5461}^{25}$ + 71° in EtOH, $[\alpha]_D$ + 34·4° in Py. Heat in vacuo with $KHSO_4$ ⟶ œstrone. Forms Me ether with diazomethane.

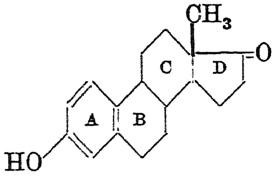
Me ether: cryst. from EtOH. M.p. 162·5–164°.

Triacetyl: m.p. 127°. Ten times as physiologically active as œstriol.

Monobenzoyl: white platelets from C_6H_6. M.p. 225°.

Cartland, Meyer, Miller, Rutz, *J. Biol. Chem.*, 1935, **109**, 215.
MacCorquodale, Thayer, Doisy, *J. Biol. Chem.*, 1933, **99**, 329.
Marrian, Haslewood, *Biochem. J.*, 1932, **26**, 25.
Butenandt, Hildebrandt, *Z. physiol. Chem.*, 1931, **199**, 245.
Marrian, *Biochem. J.*, 1930, **24**, 435, 1021.
Schwenk, Hildebrandt, Gehrke, U.S.P. 2,184,755, (*Chem. Abstracts*, 1940, **34**, 3026).

Œstrone (*Œstrin, theelin, ketohydroxyœstrin, menformon, estrone, follicular hormone, α-follicular hormone, progynon*)



$C_{18}H_{22}O_2$ MW, 270

The œstrus-producing hormone obtained, among other sources, from urine of pregnancy. Colourless cryst. from EtOH. Three polymorphic forms, m.ps. 254°, 256°, 259°. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6. Mod. sol. Et_2O, AcOEt. Spar. sol. pet. ether. Insol. H_2O. Sol. alkalis. Insol. alk. carbonates. $[\alpha]_D$ + 165° (152°, 158·5°) in $CHCl_3$. Distils undecomp. at 150–200°/0·002 mm. Does not give Liebermann–Burchard reaction. Gives no col. with $FeCl_3$.

Rings B and C, and rings C and D are *trans* fused. Heat with Pd ⟶ *d*-isoequilenin. Red. ⟶ "α"-œstradiol. *dl*-Œstrone (œstrone-b) and all the other possible *dl*-stereoisomers (a, c, d, e, f, g, h) have been obtained by total syntheses. *dl*-Œstrone has been resolved.

Me ether: $C_{19}H_{24}O_2$. MW, 284. Cryst. from EtOH. M.p. 167·5–169·5° (164–5°). $[\alpha]_D$ + 171° in $CHCl_3$. Physiologically inactive. *Semicarbazone*: cryst. from EtOH. M.p. 267° decomp.

Acetyl: needles or plates from EtOH.Aq. M.p. 123–4°. $[\alpha]_D$ + 128° in $CHCl_3$. Physiologically active.

Benzoyl: needles from MeOH. M.p. 217·5°. Physiologically active.

Oxime: $C_{18}H_{23}O_2N$. MW, 285. Needles from EtOH.Aq. Decomp. at 233°. Physiologically active.

Semicarbazone: cryst. from EtOH. M.p. 258–60° decomp. Physiologically inactive.

2 : 4-*Dinitrophenylhydrazone*: m.p. 278–80° decomp.

Œstrone-a: m.p. 214–16°. *Me ether*: m.p. 114–18°. *Benzoyl*: m.p. 175–6°.

Œstrone-b (dl-*œstrone*): m.p. 251–4° (253–5°). *Me ether*: m.p. 143–4°. *Benzoyl*: m.p. 184–6° (184–90°).

Œstrone-c: m.p. 180·5–181·5°. *Me ether*: m.p. 115–16°. *Benzoyl*: m.p. 149–51°.

Œstrone-d: m.p. 185–8°. *Me ether*: oil. *Benzoyl*: m.p. 150–2°.

Œstrone-e: m.p. 230–2°. *Me ether*: m.p. 162–4°. *Benzoyl*: m.p. 134–6°.

Œstrone-f: m.p. 191–5°. *Me ether*: oil. *Benzoyl*: m.p. 161–3°.

Œstrone-g: (dl-*lumiœstrone*): m.p. 238·5–240°. *Me ether*: m.p. 109–10°. *Benzoyl*: m.p. 157·5–158·5°.

Œstrone-h: m.p. 197–8°. *Me ether*: m.p. 67–8°. *Benzoyl*: m.p. 159·5–161·5°.

Marker, Kamm, Oakwood, Laucius, *J. Am. Chem. Soc.*, 1936, **58**, 1503.
Cartland, Meyer, Miller, Rutz, *J. Biol. Chem.*, 1935, **109**, 213.
Curtis, MacCorquodale, Thayer, Doisy, *J. Biol. Chem.*, 1934, **107**, 191.
Kofler, Hauschild, *Z. physiol. Chem.*, 1934, **224**, 150.
Butenandt, Störmer, *Z. physiol. Chem.*, 1932, **208**, 129, 149.
Doisy, Veler, Thayer, *J. Biol. Chem.*, 1930, **86**, 499; **87**, 357.
Butenandt, Ziegner, *Z. physiol. Chem.*, 1930, **188**, 1.
Johnson *et al.*, *J. Am. Chem. Soc.*, 1952, **74**, 2832.
Anner, Miescher, *Helv. Chim. Acta*, 1948, **31**, 2173; 1949, **32**, 1957; 1950, **33**, 1379.
Bachmann, Kushner, Stevenson, *J. Am. Chem. Soc.*, 1942, **64**, 974.
Dane, Schmidt, *Ann.*, 1939, **537**, 246.

Olealdehyde (*Oleic aldehyde*)

$CH_3{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}CHO$

$C_{18}H_{34}O$ MW, 266

Liq. B.p. 108–10°/0·001 mm. D_4^{20} 0·8509. n_D^{20} 1·4558.

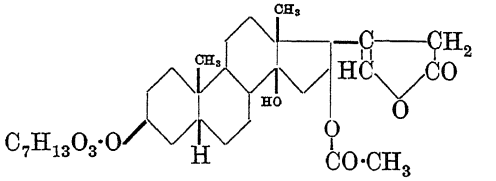
2 : 4-*Dinitrophenylhydrazone* : yellow needles from EtOH. M.p. 67–8°.

Grundmann, Bör, Trischmann, *Ann.*, 1936, **524**, 31.

Oleanan-2β-ol.
See β-Amyranol.

Oleandrigenin.
See under Gitoxigenin.

Oleandrin (*Folinerin*)

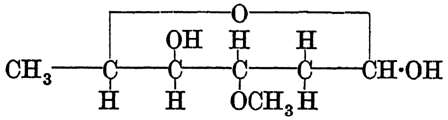


$C_{32}H_{48}O_9$ MW, 576

Glycoside from leaves of *Nerium oleander* (Laurier-rose), Linn. Compact polyhedral prismatic cryst. from EtOH, slender prisms from EtOH.Aq. M.p. 250° decomp. $[\alpha]_D^{18}$ − 52·1°. Sol. EtOH, Et_2O. Spar. sol. H_2O. Sublimes. Loses 1 mol. AcOH above m.p. ⟶ desacetyl-anhydro-oleandrin. MeOH + HCl ⟶ aglycone (oleandrigenin) which is identical with 16-acetyl-gitoxigenin.

Windaus, Westphal, *Chem. Abstracts*, 1927, **21**, 299.
Tanret, *Compt. rend.*, 1932, **194**, 914.
Kahlbaum, D.R.P. 577,257, (*Chem. Abstracts*, 1933, **27**, 4032).
Neumann, *Ber.*, 1937, **70**, 1547.
Tschesch, *ibid.*, 1554.
Hesse, *ibid.*, 2264.

Oleandrose



Probable structure

$C_7H_{14}O_4$ MW, 162

Sugar obtained by hydrolysis of oleandrin. Prisms. M.p. 68–70°. $[\alpha]_D^{18}$ − 98°.

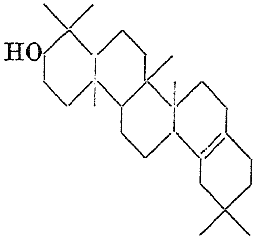
2 : 4-*Dinitrophenylhydrazone* : cryst. M.p. 155–60° decomp.

Neumann, *Ber.*, 1937, **70**, 1547.
Hess, *Ber.*, 1937, **70**, 2244.
Vischer, Reichstein, *Helv. Chim. Acta*, 1944, **27**, 1332.

Olean-12-en-2β-ol.
See β-Amyrin.

Oleanic Acid.
See Oleanolic Acid.

Oleanol



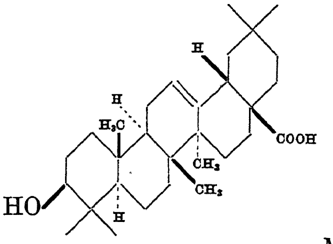
$C_{29}H_{48}O$ MW, 412

Needles from Me_2CO. M.p. 216–20°. $[\alpha]_D^{20}$ + 59° in $CHCl_3$. Gives Liebermann reaction.

Acetyl : needles from EtOH. M.p. 209–10°. $[\alpha]_D^{21}$ + 44·7° in $CHCl_3$.

Winterstein, Stein, *Z. physiol. Chem.*, 1931, **202**, 222.
Kuwada, *Chem. Abstracts*, 1936, **28**, 8237.
Barton, Brooks, *J. Am. Chem. Soc.*, 1950, **72**, 3314; *J. Chem. Soc.*, 1951, 257.

Oleanolic Acid (*Panax sapogenin, guagenin, sugar beet sapogenin, "oleanol," caryophyllin, mistletoe sapogenin, oleanic acid*)



$C_{30}H_{48}O_3$ MW, 456

Occurs as glucoside in leaves of *Aralia japonica*, (Thunb.), mistletoe, cloves, sugar beet, olive leaves, etc. Prisms from EtOH. M.p. 306–8° (305°). Sol. MeOH, EtOH. $[\alpha]_D^{12}$ + 79·5° in $CHCl_3$.

Acetyl : m.p. 268° (258–60°).

Diphenylurethane : m.p. 137–8°.

Me ester : $C_{31}H_{50}O_3$. MW, 470. M.p. 196–8° (184°). *Acetyl* : m.p. 223°. *Oxime* : m.p. 246° decomp. (226°).

Kitasato, *Chem. Abstracts*, 1934, **28**, 4051.
Aumüller, Schicke, Wedekind, *Ann.*, 1935, **517**, 211.
Ruzicka, Hofmann, *Helv. Chim. Acta*, 1936, **19**, 114.
Jacobs, Fleck, *J. Biol. Chem.*, 1932, **96**, 341.
Van der Haar, *Rec. trav. chim.*, 1927, **46**, 775, 793.
Ruzicka, Goldberg, Hofmann, *Helv. Chim. Acta*, 1937, **20**, 325.
Bischof, Jeger, Ruzicka, *Helv. Chim. Acta*, 1949, **32**, 1911.
Barton, Holness, *J. Chem. Soc.*, 1952, 78.
Klyne, *ibid.*, 2916.

Note.—The "Oleanol" described by Power, Tutin, *J. Chem. Soc.*, 1908, **93**, 896, appears to be identical with oleanolic acid.

Oleic Acid (Cis-8-*Heptadecylenecarboxylic acid*, Δ^8-*octadecylenic acid*)

$CH_3 \cdot [CH_2]_7 \cdot CH{:}CH \cdot [CH_2]_7 \cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

F.p. 11·8–12·2° (13·2°). *Labile form*: m.p. 12°. *Stable form*: m.p. 16° (15·4°). B.p. 285·5–286°/100 mm., 203–5°/5 mm., 170–5°/2–3 mm. Sol. EtOH, Et_2O. Insol. H_2O. D^{20} 0·898. n_D^{20} 1·45823 (1·4610). Heat of comb. C_p 2682·0 Cal., C_v 2677·6 Cal.

Me ester: $C_{19}H_{36}O_2$. MW, 296. B.p. 212–13°/15 mm., 160–2°/2–3 mm. D^{18} 0·879.

Et ester: $C_{20}H_{38}O_2$. MW, 310. B.p. 216–17°/151 mm. D^{25} 0·8671.

Propyl ester: $C_{21}H_{40}O_2$. MW, 324. B.p. 266–7°/15 mm.

Isopropyl ester: b.p. 223–4°/15 mm.

Butyl ester: $C_{22}H_{42}O_2$. MW, 338. B.p. 227–8°/15 mm. (235–40°/10–18 mm.).

Isobutyl ester: tebelon. B.p. 226–7°/10 mm., 190°/4 mm. D^{20} 0·86.

tert.-*Butyl ester*: b.p. 223–224·5°/10 mm.

Isoamyl ester: $C_{23}H_{44}O_2$. MW, 352. B.p. 223–4°/10 mm. D^{15} 0·897.

tert.-*Amyl ester*: b.p. 223–225·5°/10 mm.

Glyceryl esters: *see* Mono-olein.

Vinyl ester: $C_{20}H_{36}O_2$. MW, 308. B.p. 173°/2 mm.

Allyl ester: $C_{21}H_{38}O_2$. MW, 322. B.p. 219–21°/10 mm.

Phenyl ester: $C_{24}H_{38}O_2$. MW, 358. B.p. about 230°/7 mm.

m-*Tolyl ester*: $C_{25}H_{40}O_2$. MW, 372. B.p. about 240°/5 mm.

p-*Chlorophenacyl ester*: $C_{26}H_{40}O_2Cl$. MW, 419·5. M.p. 40°.

p-*Bromophenacyl ester*: $C_{26}H_{40}O_2Br$. MW, 464. M.p. 44–5°.

p-*Phenylphenacyl ester*: $C_{32}H_{45}O_2$. MW, 461. M.p. 61°.

Menthyl ester: $C_{28}H_{52}O_2$. MW, 420. B.p. about 240°/4 mm.

Stigmasteryl ester: $C_{48}H_{82}O_2$. MW, 690. M.p. 44°.

Chloride: $C_{18}H_{33}OCl$. MW, 300·5. B.p. 213°/13–15 mm., 200°/11 mm., 190°/9 mm.

Amide: $C_{18}H_{35}ON$. MW, 281. M.p. 75–6°.

Anhydride: $C_{36}H_{66}O_3$. MW, 546. M.p. 22–4°. D_4^{15} 0·900. n_D^{20} 1·4630.

Nitrile: $C_{18}H_{33}N$. MW, 263. B.p. 330–5° decomp.

Anilide: m.p. 41°. B.p. 143·5°/10 mm.

Phenylhydrazide: m.p. 72–3°.

Dibromide: see 8 : 9-Dibromostearic Acid.

Dichloride: see 8 : 9-Dichlorostearic Acid.

Noller, Bannerot, *J. Am. Chem. Soc.*, 1934, **56**, 1563.
Koyama, *Chem. Abstracts*, 1932, **26**, 5067.
Bannister, U.S.P., 1,796,231, (*Chem. Abstracts*, 1931, **25**, 2441).
Skillon, *J. Soc. Chem. Ind.*, 1931, **50**, 131T.
Raymond, *Chimie et Industrie*, Special No., 1929, 523.
Scheffers, *Rec. trav. chim.*, 1927, **46**, 293.
Bertram, *ibid.*, 397.
Holde, Gorgas, *Z. angew. Chem.*, 1926, **39**, 1443.
Robinson, Robinson, *J. Chem. Soc.*, 1925, 175.
Holde, Rietz, *Ber.*, 1924, **57**, 99.
Maihle, *Bull. soc. chim.*, 1920, **27**, 226.
Preiswerk, U.S.P., 1,318,461, (*Chem. Abstracts*, 1920, **14**, 95).
Täufel, Kunkele, *Chem. Zentr.*, 1935, I, 2971.
Brown, Shinowara, *J. Am. Chem. Soc.*, 1937, **59**, 6.
Hartsuch, *J. Am. Chem. Soc.*, 1939, **61**, 1142.

Oleic Alcohol.
See Octadecenyl Alcohol.

Oleic Aldehyde.
See Olealdehyde.

Olein.
See Triolein.

Oleodipalmitin

$C_3H_5 < \begin{matrix} O \cdot CO \cdot [CH_2]_7 \cdot CH{:}CH \cdot [CH_2]_7 \cdot CH_3 \\ (O \cdot CO \cdot [CH_2]_{14} \cdot CH_3)_2 \end{matrix}$

$C_{53}H_{100}O_6$ MW, 832

Occurs in soya bean oil, butter fat, cocoa fat, etc.

(i) M.p. 27–8°. (ii) Cryst. from Me–C_2O $CHCl_3$. M.p. 38° (37°). Remelts at 28°. (iii) M.p. 48°.

For synthetic oleodipalmitins see last three references.

Hashi, *Chem. Zentr.*, 1928, I, 1470.
Amberger, *Chem. Abstracts*, 1919, **13**, 3252.
Daubert, Fricke, Longenecker, *J. Am. Chem. Soc.*, 1943, **65**, 2143.
Jackson, Daubert, King, Longenecker, *J. Am. Chem. Soc.*, 1944, **66**, 289.
Daubert, Clarke, *ibid.*, 690.

Oleodistearin

$C_3H_5 < \begin{matrix} O \cdot CO \cdot [CH_2]_7 \cdot CH{:}CH \cdot [CH_2]_7 \cdot CH_3 \\ (O \cdot CO \cdot [CH_2]_{16} \cdot CH_3)_2 \end{matrix}$

$C_{57}H_{108}O_6$ MW, 888

Occurs in fat of seeds of *Mangifera indica*, Linn. M.p. 44° (42°, 38·5°, 29–31°). n_D^{40} 1·4570.

Amberger, Bromig, *Biochem. Z.*, 1922, **130**, 252.
Daubert, Fricke, Longenecker, *J. Am. Chem. Soc.*, 1943, **65**, 2143.

Oleone (*Diheptadecenyl ketone*)

$CH_3·[CH_2]_7·CH:CH·[CH_2]_7$
$CH_3·[CH_2]_7·CH:CH·[CH_2]_7$ >CO

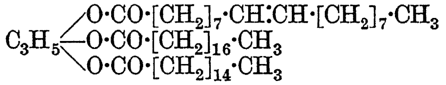
$C_{35}H_{66}O$ MW, 502

(i) Pale yellow cryst. from EtOH. M.p. 58·5°. (ii) Pale yellow oil. B.p. 120–75°/20 mm. Red. ⟶ pentatriacontane.

Oxime : m.p. 31°.

Breuer, Weinmann, *Monatsh.*, 1935, **67**, 42.
Meyer, Otto, U.S.P. 2,410,096, (*Chem. Abstracts*, 1947, **41**, 1234).

Oleopalmitostearin



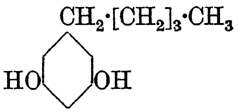
$C_{55}H_{104}O_6$ MW, 860

Occurs in palm and sardine oils.
(i) M.p. 31·3°. (ii) M.p. 42°.

Klimont, *Monatsh.*, 1902, **23**, 55.
Hansen, *Chem. Zentr.*, 1902, I, 1116.
Hilditch, Maddison, *J. Soc. Chem. Ind.*, 1940, **59**, 67.

Oleyl Alcohol.
See Octadecenyl Alcohol.

Olivetol (5-n-*Amylresorcinol*)



$C_{11}H_{16}O_2$ MW, 180

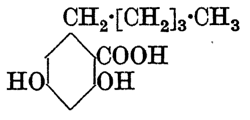
Colourless cryst. after dist. at 145° in high vacuum. M.p. 49°. Prisms + H_2O, m.p. 40–1°. Sol. ord. org. solvents. Solvated cryst. from C_6H_6-ligroin.

Di-Me ether: $C_{13}H_{20}O_2$. MW, 208. B.p. 133–6°/6 mm., 114°/2 mm. D_4^{25} 0·978. n_D^{25} 1·5056.

Di-3 : 5-*dinitrobenzoyl*: white cryst. from EtOH or Et_2O. M.p. 127–8° corr.

Asahina, Asano, *Ber.*, 1932, **65**, 478.
Asahina, Nogami, *Ber.*, 1935, **68**, 1501.
Anker, Cook, *J. Chem. Soc.*, 1945, 311.
Suter, Weston, *J. Am. Chem. Soc.*, 1939, **61**, 235.

Olivetol-carboxylic Acid (4 : 6-*Dihydroxy*-2-*amylbenzoic acid*, 6-*amyl*-β-*resorcylic acid*)



$C_{12}H_{16}O_4$ MW, 224

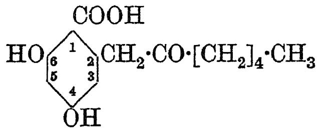
White needles. M.p. 147°.

Et ester : prisms from pet. ether. M.p. 69°.

p-*Me ether*: $C_{13}H_{18}O_4$. MW, 238. Cryst. M.p. 123–5°.

Asahina, Yasue, *Ber.*, 1937, **70**, 208.
Anker, Cook, *J. Chem. Soc.*, 1945, 811.

Olivetonic Acid (2 : 4-*Dihydroxy*-6-β-*keto-heptylbenzoic acid*)



$C_{14}H_{18}O_5$ MW, 266

Needles from hot H_2O. M.p. 159–60°. Sol. EtOH, Et_2O, Me_2CO, hot H_2O. Spar. sol. C_6H_6, ligroin.

Me ester : $C_{15}H_{20}O_5$. MW, 280. M.p. 85–6°.

4-*Me ether*: $C_{15}H_{20}O_5$. MW, 280. M.p. 94–5°.

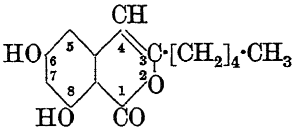
Me ester : $C_{16}H_{22}O_5$. MW, 294. M.p. 80°.

6-*Me ether* : m.p. 119–20°.

Di-Me ether : $C_{16}H_{22}O_5$. MW, 294. Needles from EtOH.Aq. M.p. 93°. *Oxime* : m.p. 120°.

Asahina, Fuzikawa, *Ber.*, 1935, **68**, 2023.
Asahina, Nogami, *Bull. Chem. Soc. Japan*, 1942, **17**, 221, (*Chem. Abstracts*, 1947, **41**, 4488).

Olivetonide



$C_{14}H_{16}O_4$ MW, 248

Needles from ligroin. M.p. 110°.

6-*Me ether*: $C_{15}H_{18}O_4$. MW, 262. Prisms from EtOH. M.p. 57°. *Acetyl*: m.p. 60–1°.

8-*Me ether* : needles from C_6H_6.. M.p. 146–7°. *Acetyl* : m.p. 84°.

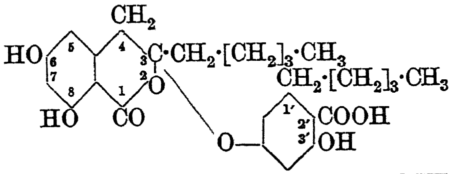
Di-Me ether: $C_{16}H_{20}O_4$. MW, 276. Prisms from EtOH. M.p. 94°.

6-*Acetyl* : m.p. 55°.

Diacetyl : m.p. 59°.

Asahina, Fuzikawa, *Ber.*, 1935, **68**, 82, 2023.
Asahina, Nogami, *Bull. Chem. Soc. Japan*, 1942, **17**, 221, (*Chem. Abstracts*, 1947, **41**, 4488).

Olivetoric Acid



$C_{26}H_{32}O_8$ MW, 472

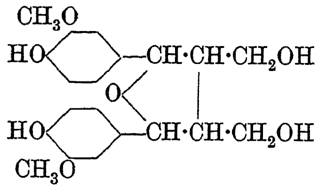
Occurs in lichens *Alectoria divergens* Nyl, *Parmelia olivetorum* Nyl, *Evernia olivetorina* Zopf. Cryst. from hot C_6H_6. M.p. 151°. Sol. EtOH, Et_2O, Me_2CO, AcOH. Spar. sol. C_6H_6. Insol. ligroin.

6 : 3′-*Di-Me ether*: *Me ester*, $C_{29}H_{38}O_8$. MW, 514. M p. 123°.

Me ester: $C_{27}H_{34}O_8$. MW, 486. Needles. M.p. 134°.

Asahina, Fuzikawa, *Ber.*, 1935, **68**, 2026.
Asahina, Asano, *Ber.*, 1932, **65**, 584.
See also Hesse, *J. prakt. Chem.*, 1916, **94**, 227.

Olivil



$C_{20}H_{24}O_7$ MW, 376

Occurs in olive-wood resin. Cryst. + $1H_2O$ from H_2O. M.p. 105°, anhyd. 142·5°. $[\alpha]_D^{12}$ − 127°.

Me ether: $C_{21}H_{26}O_7$. MW, 390. M.p. 238°.

Di-Me ether: $C_{22}H_{28}O_7$. MW, 404. M.p. 156°.

Et ether: $C_{22}H_{28}O_7$. MW, 404. M.p. 145°.

Di-Et ether: $C_{24}H_{32}O_7$. MW, 432. M.p. 182°.

Me-Et ether: $C_{23}H_{30}O_7$. MW, 418. M.p. 169°.

Dipropyl ether: $C_{26}H_{36}O_7$. MW, 460. M.p. 135·5°.

Vanzetti, Dreyfuss, *Gazz. chim. ital.*, 1934, **64**, 381.
Dreyfuss, *Gazz. chim. ital.*, 1936, **66**, 96.
Atkinson, Haworth, *J. Chem. Soc.*, 1938, 1683.
Vanzetti, Dreyfuss, *Chem. Abstracts*, 1937, **31**, 7865.

Onocerin.
See Onocerol.

Onocerol (*Onocol, onocerin*)

$C_{30}H_{50}O_2$ MW, 442

A triterpene occurring in *Ononis spinosa*, Linn. Prisms from EtOH. M.p. 232°. Sol. isoamyl alcohol. Spar. sol. Et_2O, $CHCl_3$, AcOEt. Insol. H_2O. Sublimes. $[\alpha]_D^{20}$ + 12·05°.

Diacetyl deriv.: m.p. 224°. $[\alpha]_D^{18}$ + 28·3° in $CHCl_3$.

Di-chloroacetyl deriv.: m.p. 238–44°. $[\alpha]_D^{19}$ + 115° in $CHCl_3$.

Dibenzoyl deriv.: m.p. 237–8°. $[\alpha]_D^{22}$ + 21·3° in $CHCl_3$.

*Di-*3 : 5-*dinitrobenzoyl deriv.*: m.p. 290–1°. $[\alpha]_D^{19}$ + 19·3° in $CHCl_3$.

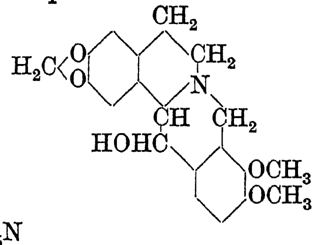
Dianisoyl deriv.: m.p. 232–4°. $[\alpha]_D^{22}$ + 10° in $CHCl_3$.

Schulze, *Z. physiol. Chem.*, 1936, **238**, 35.
Dieterle, Salomon, Gärtner, *Chem. Abstracts*, 1934, **28**, 4065.
Zimmermann, *Helv. Chim. Acta*, 1938, **21**, 853; 1940, **23**, 1110.

Onocol.
See Onocerol.

Oöcyan.
See Biliverdin.

Ophiocarpine

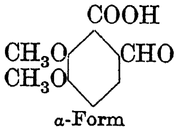


$C_{20}H_{21}O_5N$ MW, 355

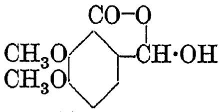
Principal alkaloidal constituent (0·25%) of *Corydalis ophiocarpa*. M.p. 188°. $[\alpha]_D^{24}$ − 284° in $CHCl_3$.

Manske, *Chem. Abstracts*, 1939, **33**, 6321.

Opianic Acid (5 : 6-*Dimethoxy*-o-*aldehydobenzoic acid*, 6-*aldehydo*-o-*veratric acid*, 5 : 6-*dimethoxy*-o-*phthalaldehydic acid*)



α-Form



ψ-Form

$C_{10}H_{10}O_5$ MW, 210

Prisms from H_2O. M.p. 150° (146°). Sol. to 120 gm. in 100 ml. hot H_2O. Present in α-form in aq. sol. and dil. alkalis. Dissolves in EtOH gradually forming the ψ-Et ester. Slightly sol. Et_2O. Present in ψ-form in $CHCl_3$. Mixture of α- and ψ-forms present in 96% EtOH. $k = 8{\cdot}82 \times 10^{-4}$ at 25°. Ox. ⟶ hemipinic acid. Red. ⟶ meconin.

α-Me ester: $C_{11}H_{12}O_5$. MW, 224. Needles from EtOH. M.p. 82–3°. B.p. 232–4°/52 mm. *Diacetyl*: m.p. 88–9°. *Semicarbazone*: m.p. 204°.

ψ-Me ester: m.p. 105°.

α-Et ester: $C_{12}H_{14}O_5$. MW, 238. M.p. 64°.

ψ-Et ester: m.p. 92–3°.

ψ-Propyl ester: $C_{13}H_{16}O_5$. MW, 252. M.p. 103°.

ψ-tert.-*Amyl ester*: $C_{15}H_{20}O_5$. MW, 280. M.p. 81°.

α-Benzyl ester: $C_{17}H_{16}O_5$. MW, 300. M.p. 82–3°.

ψ-Benzyl ester: m.p. 94–5°.

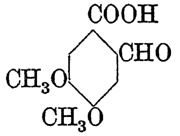
ψ-Chloride: $C_{10}H_9O_4Cl$. MW, 228·5. Needles from C_6H_6. M.p. 93–4°.

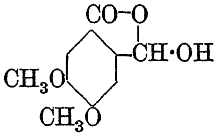
Anhydride: $C_{20}H_{18}O_9$. MW, 402. M.p. 234°.

Oxime: needles. M.p. 82°.
Semicarbazone: m.p. 187°.
Benzoylhydrazone: m.p. 227° decomp.
Anil: cryst. from EtOH. M.p. 191·5°–192·5°.

Kanewskaja *et al.*, *Ber.*, 1936, **69**, 257.
Edwards, Perkin, Stoyle, *J. Chem. Soc.*, 1925, 197.
Kirpal, *Ber.*, 1927, **60**, 382.
Schorigin, Issagulanz, Below, *Ber.*, 1931, **64**, 1931.
Wegscheider, Späth, *Monatsh.*, 1916, **37**, 277.
Rodionow, Fedorova, *Ber.*, 1926, **59**, 2949.
Buu-Hoï, *Compt. rend.*, 1941, **212**, 242.

m-Opianic Acid (4 : 5-*Dimethoxy*-o-*aldehydobenzoic acid*, 6-*aldehydoveratric acid*, 4 : 5-*dimethoxy*-o-*phthalaldehydic acid*)


α-Form

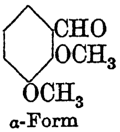

ψ-Form

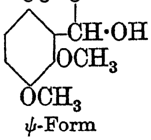
$C_{10}H_{10}O_5$ MW, 210

Needles from H_2O. M.p. 185°. Sol. AcOH, hot Me_2CO. Spar. sol. H_2O, MeOH.
α-Me ester: $C_{11}H_{12}O_5$. MW, 224. M.p. 93–5°. *Anil*: m.p. about 143°.
ψ-Me ester: m.p. 142–3°.
Anhydride: $C_{20}H_{18}O_9$. MW, 402. M.p. about 230°.
Oxime: m.p. about 140°.
Semicarbazone: m.p. about 227°.
Phenylhydrazone: m.p. 228°.

Vanzetti, Oliverio, Cavinato, *Gazz. chim. ital.*, 1931, **61**, 479.
Perkin, Stoyle, *J. Chem. Soc.*, 1923, **123**, 3171.

ψ-Opianic Acid (3 : 4-*Dimethoxy*-o-*aldehydobenzoic acid*, 2-*aldehydoveratric acid*, 3 : 4-*dimethoxy*-o-*phthalaldehydic acid*)


α-Form

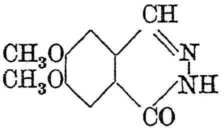

ψ-Form

$C_{10}H_{10}O_5$ MW, 210

Needles from H_2O or C_6H_6. M.p. 121–2°. Sol. hot H_2O, EtOH, $CHCl_3$, C_6H_6. Spar. sol. pet. ether. Hot conc. KOH.Aq. ⟶ veratric acid.
Oxime: needles from H_2O. M.p. 124° decomp.

Perkin, *J. Chem. Soc.*, 1890, **57**, 1064.
Chakravarti, Swaminathan, *Chem. Abstracts*, 1934, **28**, 6720.

Opiazone (6 : 7-*Dimethoxyphthalazone*)



$C_{10}H_{10}O_3N_2$ MW, 206

Needles. M.p. anhyd. 166°. Sol. EtOH, AcOH, hot H_2O.
N-*Acetyl*: m.p. 158–9°.

Liebermann, Bistrzycki, *Ber.*, 1893, **26**, 532.

Opsopyrrole.
See 4-Methyl-3-ethylpyrrole.

Optochine (*Dihydrocupreine ethyl ether*)

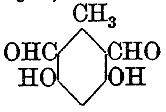
$C_{21}H_{28}O_2N_2$ MW, 340

M.p. 80–4° (contains H_2O and toluene). M.p. 123–8° (solvent free). Sol. ord. org. solvents. Mod. sol. C_6H_6, toluene, ligroin. $k_1 = 3{\cdot}2 \times 10^{-6}$, $k_2 = 1{\cdot}1 \times 10^{-10}$ in H_2O at 23°. $[\alpha]_D^{26\cdot5} - 112{\cdot}7°$ in EtOH ($[\alpha]_D^{25} - 136{\cdot}2°$ in EtOH).
Dimethiodide: yellow needles + $2H_2O$ from H_2O. M.p. 233–5°.

Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1922, **44**, 1097.
Kaishi, *Chem. Abstracts*, 1947, **41**, 4582.

Orcacetophenone.
See 4 : 6-Dihydroxy-2-methylacetophenone.

α-Orcindialdehyde (4 : 6-*Dihydroxy*-2-*methylisophthaldehyde*)

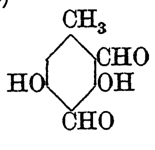


$C_9H_8O_4$ MW, 180

Needles from hot H_2O. M.p. 117–19°. Sol. EtOH, Et_2O, $CHCl_3$. Mod. sol. hot H_2O. Volatile in steam.

Tiemann, Helkenberg, *Ber.*, 1879, **12**, 1003.

β-Orcindialdehyde (2 : 6-*Dihydroxy*-4-*methylisophthalaldehyde*)

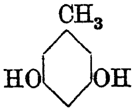


$C_9H_8O_4$ MW, 180

Cryst. from EtOH.Aq. M.p. 168°. Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O.

Tiemann, Helkenberg, *Ber.*, 1879, **12**, 1004.

Orcinol (5-*Methylresorcinol*, 3 : 5-*dihydroxytoluene*)



$C_7H_8O_2$ MW, 124

Cryst. + $1H_2O$ from H_2O, leaflets from $CHCl_3$. (i) M.p. 106·5–108°. (ii) M.p. 107·5°. B.p. 287–90°. Sol. H_2O, EtOH, Et_2O, C_6H_6. Spar. sol. ligroin, pet. ether. Heat of comb. C_p 824·72 Cal.

Diacetyl: m.p. 25°. Fries rearrangement ⟶ 2 : 4-diacetylorcinol.

Dibenzoyl: m.p. 88°.

Me ether: $C_8H_{10}O_2$. MW, 138. M.p. 61–2°. B.p. 259°/755 mm., 130°/6·5 mm. D^{15} 1·1106. n_D^{20} 1·54734.

Di-Me ether: $C_9H_{12}O_2$. MW, 152. B.p. 244°, 102°/8 mm. D^{15} 1·0478. n_D^{29} 1·52342.

Et ether: $C_9H_{12}O_2$. MW, 152. B.p. 265–70°.

Di-Et ether: $C_{11}H_{16}O_2$. MW, 180. M.p. 16–16·5°. B.p. 251–2°/747·5 mm.

Picrate: m.p. 92°.

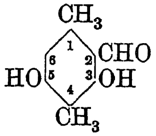
Schaum, *Ann.*, 1928, **462**, 207.
Missenden, *Chemical Age*, 1922, **7**, 709 (*Review*).
Walbaum, Rosenthal, *Ber.*, 1924, **57**, 771.
Vogt, Henninger, *Bull. soc. chim.*, 1874, **21**, 273.
Anker, Cook, *J. Chem. Soc.*, 1945, 312.

β-Orcinol.
See 2 : 6-Dihydroxy-*p*-xylene.

γ-Orcinol.
See 2 : 4-Dihydroxytoluene.

Orcinol-carboxylic Acid.
See Orsellinic Acid *and* 3 : 5-Dihydroxy-*p*-toluic Acid.

β-Orcylaldehyde (β-*Orcinaldehyde*, 3 : 5-*dihydroxy*-4-*methyl*-o-*toluic aldehyde*, 4 : 6-*dihydroxy*-2 : 5-*dimethylbenzaldehyde*)



$C_9H_{10}O_3$ MW, 166

Needles from EtOH. M.p. 166°.

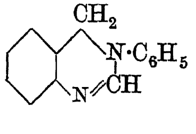
5-*Me ether*: *see* Rhizonaldehyde.

Sonn, *Ber.*, 1931, **64**, 185.
Robertson, Stephenson, *J. Chem. Soc.*, 1930, 316.

γ-Orcylaldehyde.
See Atranol.

Orcylic Aldehyde.
See 3 : 5-Dihydroxy-*o*-toluic Aldehyde.

Orexine (3-*Phenyl*-3 : 4-*dihydroquinazoline*)



$C_{14}H_{12}N_2$ MW, 208

Plates from Et_2O–ligroin. M.p. 94–6°. Sol. EtOH, Et_2O, $CHCl_3$. Insol. H_2O.

B,HCl,2H₂O: m.p. 80°, anhyd. 221°.

B,HCl,SnCl₂: m.p. 130–4°.

B₂,H₂PtCl₆: m.p. 208°.

B₂,H₂SO₄,2H₂O: m.p. 70°, anhyd. 140–3°.

Methiodide: m.p. 170°.

Paal, Busch, *Ber.*, 1889, **22**, 2686.
Kalle, D.R.P., 113,163, (*Chem. Zentr.*, 1900, II, 615).

Oripavine

$C_{18}H_{21}O_3N$ MW, 299

Alkaloid from *Papaver orientalis* and *Papaver bracteatum*. Needles from EtOH. M.p. 201–2° (200–1°). Sol. $CHCl_3$. Spar. sol. EtOH, Me_2CO. Insol. H_2O.

B,HCl: cryst. from EtOH. M.p. 258–9° decomp. (244–5°).

Methiodide: m.p. 207–8°.

Konowalowa, Yunussoff, Orechoff, *Ber.*, 1935, **68**, 2160.
Kiselev, Konovalova, *J. Gen. Chem. U.S.S.R.*, 1948, **18**, 142, (*Chem. Abstracts*, 1948, **42**, 5037).

Orixidine

$C_{15}H_{13}O_4N$ MW, 271

Needles from + $1\frac{1}{2}H_2O$, EtOH.Aq. M.p. 195°. Cryst. (+ 1AcOH ?) from AcOH. M.p. 142°.

Terasaka, *Chem. Abstracts*, 1932, **26**, 730.

Orixine

$C_{18}H_{23}O_6N$ ($C_{18}H_{21}O_6N$) MW, 349 (347)

Alkaloid from root of *Orixa japonica*, Thunb. M.p. 152·5°. Sol. EtOH, Et_2O, $CHCl_3$, AcOEt. Insol. pet. ether. $[\alpha]_D^{17}$ + 83·29° in $CHCl_3$.

B,HAuCl₄: decomp. at 155°.

Terasaka, *Chem. Abstracts*, 1932, **26**, 730.

Orizabin.
See Jalapin.

Ornithine (1 : 4-*Diamino*-n-*valeric acid*)

$H_2N{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$

$C_5H_{12}O_2N_2$ MW, 132

d-.

M.p. 140°. Sol. H_2O, EtOH. Spar. sol. Et_2O. $[\alpha]_D^{25}$ + 11·5°.

B,2HCl: $[\alpha]_D$ + 16·8° in H_2O.

B,H₂PtCl₆: decomp. at 200–10°.

Monosulphate: decomp. at 234°.

Acetate: m.p. 161–2°.

1 : 4-N-*Dibenzoyl*: *see* Ornithuric Acid.

Dipicrate: decomp. at 208°.

dl-.

B,HCl: m.p. 215°.

B,HNO₃: m.p. 183°.

B,H₂SO₄: decomp. at 208°.

B₂,H₂SO₄: white plates from 60% EtOH. M.p. 223° decomp. (213°, 234°).

Acetate: m.p. 163–4°.

Oxalate: m.p. 218°.

Picrolonate : m.p. 220–1° decomp.
Dipicrolonate : m.p. 235–6°.
4-N-*Benzoyl* : m.p. 285–8°.
Dipicrate : decomp. at 208° (m.p. 195°).
1-N-*Me* : 4-amino-1-methylamino-*n*-valeric acid. $C_6H_{14}O_2N_2$. MW, 146. *B,HAuCl$_4$* : needles. M.p. 130–2°. *Flavianate* : m.p. 222–3°. 4-N-*Benzoyl*-1-N-p-*toluenesulphonyl* : needles from 70% EtOH. M.p. 185°. 1-N-p-*Toluenesulphonyl* : plates from H_2O. M.p. 214–19°. Prac. insol. EtOH, Et_2O, ligroin.
4-N-*Me* : 1-amino-4-methylamino-*n*-valeric acid. *B,HCl* : m.p. about 215–25°. *B,2HCl* : m.p. 157°. Very sol. H_2O. 4-N-*Benzoyl* : m.p. 215°. Sol. H_2O. 1-N-p-*toluenesulphonyl* : decomp. at 245°. 4-N-*Benzoyl*-1-N-p-*toluenesulphonyl* : needles from AcOH. M.p. 188–9°. *Picrate* : decomp. at 205–6°.

Albertson, Archer, *J. Am. Chem. Soc.*, 1945, **67**, 2043.
Adamson, *J. Chem. Soc.*, 1939, 1567.
Hunter, *Biochem. J.*, 1939, **33**, 29.
Kurtz, *J. Biol. Chem.*, 1938, **122**, 480.

Ornithuric Acid (1 : 4-N-*Dibenzoylornithine*)

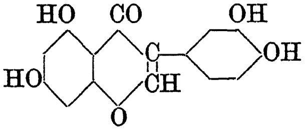
$C_6H_5\cdot CO\cdot NH\cdot[CH_2]_3\cdot CH(NH\cdot CO\cdot C_6H_5)\cdot COOH$

$C_{19}H_{20}O_4N_2$ MW, 340

d-.
Needles or plates from EtOH. M.p. 188–9°. Sol. AcOEt, hot EtOH. Spar. sol. H_2O. Insol. Et_2O. $[\alpha]_D^{20}$ + 8·5° in EtOH.Aq.
Me ester : $C_{20}H_{22}O_4N_2$. MW, 354. M.p. 145–6°.
Et ester : $C_{21}H_{24}O_4N_2$. MW, 368. M.p. 155°.
l-.
M.p. 189°. $[\alpha]_D^{20}$ − 9·22° in NaOH.Aq.
dl-.
Needles from EtOH. M.p. 187–8°.

Karrer, Escher, Widmer, *Helv. Chim. Acta*, 1926, **9**, 301.
Boon, Robson, *Biochem. J.*, 1935, **29**, 2684.

Orobol (5 : 7 : 3′ : 4′-*Tetrahydroxyisoflavone*)



$C_{15}H_{10}O_6$ MW, 286

Occurs as glucoside, oroboside, in *Orobus tuberosus*, L. Pale yellow needles from AcOH. M.p. 270·5°. 30% KOH ⟶ phloroglucinol + homoprotocatechuic acid.
Tetra-acetyl : cryst. from AcOH. M.p. 210–12°.

Bridel, Charaux, *Compt. rend.*, 1930, **190**, 387; *Bull. soc. chim. biol.*, 1930, **12**, 615.
Charaux, Rabaté, *Bull. soc. chim. biol.*, 1939, **21**, 1330.

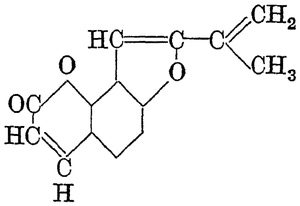
Oroboside

$C_{21}H_{20}O_{11}$ MW, 448

Occurs in *Orobus tuberosus*, Linn. Yellow prisms from 40% EtOH.Aq. M.p. 220–1°. $[\alpha]_D$ − 61·3° in Py. Acid hyd. ⟶ orobol + glucose.

Bridel, Charaux, *Compt. rend.*, 1930, **190**, 387; *Bull. soc. chim. biol.*, 1930, **12**, 615.

Oroselone

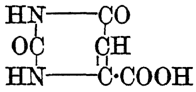


$C_{14}H_{10}O_3$ MW, 226

Obtained by acid treatment of athamantin (extract of *Athamanta oreoselinum*, Linn.). Cryst. M.p. 188–9°.

Späth, Platzer, Schmid, *Ber.*, 1940, **73**, 709, 1309.

Orotic Acid (*Uracil*-4-*carboxylic acid*)

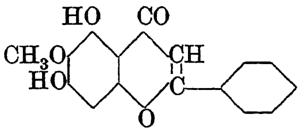


$C_5H_4O_4N_2$ MW, 156

Occurs in milk. Cryst. from H_2O. M.p. 345–6°. H_2O_2 + conc. HCl ⟶ 5-chlorouracil-4-carboxylic acid.
Me ester : $C_6H_6O_4N_2$. MW, 170. M.p. 249°.

Bachstez, *Ber.*, 1931, **64**, 2683.
Hilbert, *J. Am. Chem. Soc.*, 1932, **54**, 2082.
Johnson, Schroeder, *J. Am. Chem. Soc.*, 1932, **54**, 2942.
Mitchell, Nyc, *J. Am. Chem. Soc.*, 1947, **69**, 674, 1382.

Oroxylin-A (5 : 7-*Dihydroxy*-6-*methoxyflavone*)



$C_{16}H_{12}O_5$ MW, 284

Colouring matter of root bark of *Oroxylum indicum*, Vent. Yellow needles from EtOH. M.p. 231–2°.
Diacetyl : m.p. 131–2°.
7-*Benzoyl* : m.p. 210°.
7-*Me ether* : $C_{17}H_{14}O_5$. MW, 298. M.p. 155–6°. *Platinichloride* : m.p. 185–7° decomp. 5-*Acetyl* : m.p. 130–1°. 5-*Benzoyl* : m.p. 206–7°.
5 : 7-*Di-Me ether* : $C_{18}H_{16}O_5$. MW, 312. M.p. 165–6°.

5 : 7-*Di-Et ether* : m.p. 115–16°.

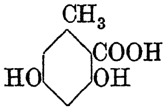
Shah, Mehta, Wheeler, *J. Chem. Soc.*, 1936, 591.

Sastri, Seshadri, *Current Science*, 1946, **15**, 235, (*Chem. Abstracts*, 1947, **41**, 2417).

Orsellic Acid.
See Orsellinic Acid.

***p*-Orsellinaldehyde.**
See Atranol.

Orsellinic Acid (*Orsellic acid*, 3 : 5-*dihydroxy-o-toluic acid*, *orcinol-2-carboxylic acid*, 6-*methyl-β-resorcylic acid*)



$C_8H_8O_4$ MW, 168

Needles + $1H_2O$ from AcOH.Aq. M.p. 176° decomp. Sol. EtOH, Et_2O. $k = 1{\cdot}271 \times 10^{-4}$ at 25°.

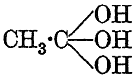
Me ester : $C_9H_{10}O_4$. MW, 182. M.p. 140°.
Et ester : $C_{10}H_{12}O_4$. MW, 196. M.p. 132°.
Isoamyl ester : $C_{13}H_{18}O_4$. MW, 238. M.p. 76°.
Diacetyl : m.p. 142°. *Chloride* : m.p. 56–8°.
3-*Me ether* : *see* Isoeverninic Acid.
5-*Me ether* : *see* Everninic Acid.
Di-Me ether : *see under* Everninic Acid.
5-*Me*-3-*Et ether* : *see under* Everninic Acid.

Hesse, *Ann.*, 1866, **139**, 35.
Hoesch, *Ber.*, 1913, **46**, 888.
Koller, *Monatsh.*, 1932, **61**, 147.
Sonn, *Ber.*, 1928, **61**, 926.

***p*-Orsellinic Acid.**
See 3 : 5-Dihydroxy-*p*-toluic Acid.

Orthanilic Acid.
See Aniline-*o*-sulphonic Acid.

Orthoacetic Acid (1 : 1 : 1-*Trihydroxyethane*)

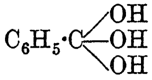


$C_2H_6O_3$ MW, 78

Tri-Me ester : $C_5H_{12}O_3$. MW, 120. B.p. 107–9°. D_4^{25} 0·94375. n_D^{25} 1·38585.
Di-Me : *Et ester* : $C_6H_{14}O_3$. MW, 134. B.p. 123–6°. D_4^{25} 0·91915. n_D^{25} 1·38885.
Me-di-Et ester : $C_7H_{16}O_3$. MW, 148. B.p. 135–6°. D_4^{25} 0·90085. n_D^{25} 1·39185.
Me-di-isobutyl ester : $C_7H_{16}O_3$. MW, 148. B.p. 205–6°.
Me-di-isoamyl ester : $C_9H_{20}O_3$. MW, 176. B.p. 219–23°.
Tri-Et ester : $C_8H_{18}O_3$. MW, 162. B.p. 144–6°. D_4^{25} 0·8847. n_D^{25} 1·39485.
Et-dipropyl ester : $C_{10}H_{22}O_3$. MW, 190. B.p. 190–4°. D_4^{25} 0·87129. n_D^{25} 1·40635.
Et-di-butyl ester : $C_{12}H_{26}O_3$. MW, 218. B.p. 220–5°. D_4^{25} 0·86461. n_D^{25} 1·41485.
Di-isobutyl ester : $C_{10}H_{22}O_3$. MW, 190. B.p. 207–8°.
Tri-isobutyl ester : $C_{14}H_{30}O_3$. MW, 246. B.p. 217°.
Triphenyl ester : white cryst. from EtOH.Aq. M.p. 61–2°. B.p. 148–53°/0·5 mm. N/10 HCl ⟶ phenol + phenyl acetate.

Sah, *J. Am. Chem. Soc.*, 1928, **50**, 516; *Chem. Abstracts*, 1933, **27**, 5729.
Sigmund, Herschdörfer, *Monatsh.*, 1931, **58**, 284.
McElvain, Nelson, *J. Am. Chem. Soc.*, 1942, **64**, 1827.
McElvain, Burkett, *ibid.*, 1833.
McElvain, Fajardo-Pinzón, *J. Am. Chem. Soc.*, 1945, **67**, 652.

Orthobenzoic Acid (ω-*Trihydroxytoluene*)

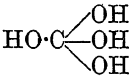


$C_7H_8O_3$ MW, 140

Tri-Me ester : $C_{10}H_{14}O_3$. MW, 182. B.p. 183–99°, 90°/35 mm.
Tri-Et ester : $C_{13}H_{20}O_3$. MW, 224. B.p. 238–40°/747 mm., 130–5°/24 mm. D_0^{20} 0·9902. Slowly decomp. when refluxed ⟶ ethyl benzoate + Et_2O.
Di-2-naphthyl ester anhydride : needles from $PhNO_2$. Does not melt below 350°.

Doebner, *Ann.*, 1890, **257**, 59.
Tschitschibabin, *Ber.*, 1905, **38**, 563.
Vila, Jarque, *Chem. Abstracts*, 1945, **39**, 3784.

Orthocarbonic Acid (*Tetrahydroxymethane*)

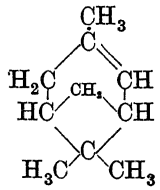


CH_4O_4 MW, 80

Tetra-Me ester : $C_5H_{12}O_4$. MW, 136. M.p. − 5·5°. B.p. 114°. $D_{18.5}^{18.5}$ 1·0232. n_D^{16} 1·3864.
Tetra-Et ester : $C_9H_{20}O_4$. MW, 192. B.p. 158–9°, 62°/28 mm. $D_4^{18.5}$ 0·9197. $n_D^{18.5}$ 1·39354.
Tetrapropyl ester : $C_{13}H_{28}O_4$. MW, 248. B.p. 224·2°.
Tetra-isobutyl ester : $C_{17}H_{36}O_4$. MW, 304. B.p. 244·9°.

Hartel, *Ber.*, 1927, **60**, 1841.
Röse, *Ann.*, 1880, **205**, 250.
Wilke, *Z. anorg. allgem. Chem.*, 1921, **119**, 377.
Connolly, Dyson, *J. Chem. Soc.*, 1937, 828.

Orthodene



$C_{10}H_{16}$ MW, 136

Occurs in essential oil of *Orthodon lanceolatum*, Kudo, and in leaf oil of camphor seedlings (but not in leaf oil of mature camphor tree). B.p. 168–70°/757 mm. D_4^{20} 0·8430. n_D^{20} 1·4670. $[\alpha]_D^{29}$ + 32·6°.

Fujita, *Chem. Abstracts*, 1934, **28**, 1470.
Guha, Narasimha Rao, *Ber.*, 1938, **71**, 1591.

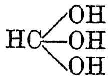
Orthodonene

$C_{15}H_{24}$ MW, 204

Occurs in essential oil of *Orthodon lanceolatum*, Kudo. B.p. 254°/700 mm. D_4^{30} 0·9017. n_D^{30} 1·4947. $[\alpha]_D^{14}$ − 13·28°.

Fujita, *Chem. Abstracts*, 1934, **28**, 1470.

Orthoformic Acid (*Trihydroxymethane*)



CH_4O_3 MW, 64

Tri-Me ester: $C_4H_{10}O_3$. MW, 106. B.p. 103–5°. D_4^{20} 0·9676. n_D^{20} 1·3793.

Tri-Et ester: $C_7H_{16}O_3$. MW, 148. B.p. 145–7°, 60°/30 mm. D_4^{20} 0·8909. n_D^{20} 1·3922. $Ac_2O \longrightarrow$ AcOEt + diethoxymethyl acetate.

Di-Et: propyl ester: $C_8H_{18}O_3$. MW, 162. B.p. 165°/747 mm., 81°/30 mm. D_4^{23} 0·8813. n_D^{20} 1·3989.

Et-dipropyl ester: $C_9H_{20}O_3$. MW, 176. B.p. 184°/745 mm., 93°/30 mm. D_4^{22} 0·8973. n_D^{20} 1·4031.

Tripropyl ester: $C_{10}H_{22}O_3$. MW, 190. B.p. 196–8°, 190–1°/745 mm., 93°/30 mm. D_4^{20} 0·8805. n_D^{20} 1·4072.

Dipropyl-isoamyl ester: $C_{12}H_{26}O_3$. MW, 218. B.p. 124–30°/24 mm. D_4^{23} 0·8647. n_D^{20} 1·415.

Propyl-di-isoamyl ester: $C_{14}H_{30}O_3$. MW, 246. B.p. 140–7°/30 mm. D_4^{23} 0·8626. n_D^{20} 1·4194.

Tri-isopropyl ester: b.p. 166–8°. D_4^{20} 0·8621. n_D^{20} 1·4000.

Tributyl ester: $C_{13}H_{28}O_3$. MW, 232. B.p. 245–7°. D_4^{20} 0·8693. n_D^{20} 1·4180.

Tri-isobutyl ester: b.p. 224–6°. D_4^{20} 0·8582. n_D^{20} 1·4120.

Tri-isoamyl ester: $C_{16}H_{34}O_3$. MW, 274. B.p. 267–9°, 166°/25 mm. D_4^{20} 0·8628. n_D^{20} 1·4233.

Triphenyl ester: $C_{19}H_{16}O_3$. MW, 292. M.p. 76–7°. B.p. 269–70°/50–5 mm. decomp. Sol. Et_2O, $CHCl_3$, hot EtOH, hot C_6H_6.

*Tri-*o*-tolyl ester*: $C_{22}H_{22}O_3$. MW, 334. M.p. 96°.

*Tri-*m*-tolyl ester*: m.p. 50°.

*Tri-*p*-tolyl ester*: m.p. 112°.

Trianilide: trianilinomethane. M.p. 138°. *B,3HCl*: m.p. 240° decomp.

*Tri-*o*-toluidide*: m.p. 150–1°. *B,3HCl*: m.p. 212–13°.

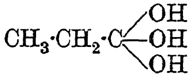
*Tri-*m*-toluidide*: m.p. 123°. *B,3HCl*: m.p. 221–2°.

Giacalone, *Gazz. chim. ital.*, 1932, **62**, 577.
Post, Erickson, *J. Am. Chem. Soc.*, 1933, **55**, 3851 (*Bibl.*).
Driver, *J. Am. Chem. Soc.*, 1924, **46**, 2090.
Hunter, *J. Chem. Soc.*, 1924, **125**, 1392.
Monchel, Agre, Hanford, *J. Am. Chem. Soc.*, 1948, **70**, 2268.
Chu, Shen, *J. Chinese Chem. Soc.*, 1943, **10**, 124, (*Chem. Abstracts*, 1944, **38**, 2930).

Orthoform New.
See under 4-Hydroxy-*m*-aminobenzoic Acid.

Orthoform Old.
See under 3-Hydroxy-*p*-aminobenzoic Acid.

Orthopropionic Acid (1 : 1 : 1-*Trihydroxypropane*)



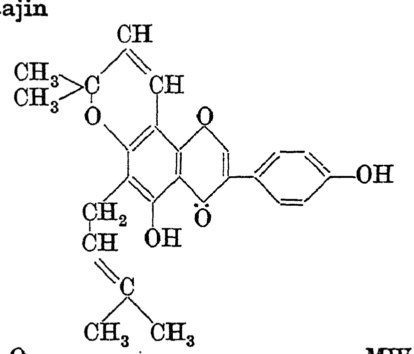
$C_3H_8O_3$ MW, 92

Tri-Et ester: $C_9H_{20}O_3$. MW, 176. B.p. 159–60°, 44°/9 mm. n_D^{25} 1·4000.

Kodak-Pathé, F.P., 712,995, (*Chem. Abstracts*, 1932, **26**, 1531).
Sigmund, Herschdörfer, *Monatsh.*, 1931, **58**, 282.
McElvain, Nelson, *J. Am. Chem. Soc.*, 1942, **64**, 1827.

Oryzanin.
See Aneurin.

Osajin



$C_{25}H_{24}O_5$ MW, 404

Isolated from fruit of osage orange tree (*Maclura pomifera*, Raf.). Lemon yellow cryst. from xylene or 95% EtOH. M.p. 189° (193° corr.). Very sol. $CHCl_3$, Et_2O, Me_2CO, Py. Mod. sol. C_6H_6, warm CCl_4. Prac. insol. H_2O, pet. ether. Reduces Fehling's, and Tollen's in Py. Alc. sol. + aq. $FeCl_3 \longrightarrow$ dark green col. $\longrightarrow$ reddish violet on addn. of a few drops of NH_4OH.

Monoacetyl deriv.: yellow cryst. from EtOH. M.p. 159°.

Diacetyl deriv.: cryst. from EtOH.Aq. Two forms. M.ps. 162° and 152°.

*Mono-*p*-toluenesulphonyl*: yellow plates from EtOH. M.p. 152°.

Me ether: $C_{26}H_{26}O_5$. MW, 418. Yellow needles from 95% EtOH. M.p. 134–5°. *Mono-acetyl*: prisms from 95% EtOH. M.p. 140–140·5°.

Di-Me ether: $C_{27}H_{28}O_5$. MW, 432. Needles from 95% EtOH. M.p. 118·5°.

Wolfrom, Gregory, *J. Am. Chem. Soc.*, 1940, **62**, 651.

Wolfrom, Benton, Gregory, Hess, Mahan, Morgan, *J. Am. Chem. Soc.*, 1939, **61**, 2833.

Walter, Wolfrom, Hess, *J. Am. Chem. Soc.*, 1938, **60**, 575.

Wolfrom *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 406.

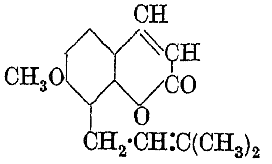
Oscine.

See Scopoline.

Osotriazole.

See 1 : 2 : 3-Triazole.

Osthol



$C_{15}H_{16}O_3$ MW, 244

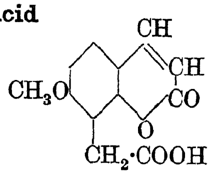
Occurs in root of *Imperatoria ostruthium*, Linn. Needles from EtOH.Aq. M.p. 83–4° (85°). B.p. 145–50°. Sol. EtOH, MeOH, $CHCl_3$, Me_2CO, AcOEt. Insol. H_2O, pet. ether.

Späth, Holzen, *Ber.*, 1934, **67**, 264.

Yamashita, *Bull. Chem. Soc. Japan*, 1933, **8**, 276.

Eiter, Meinhard, *Ber.*, 1942, **75**, 1623.

Ostholic Acid



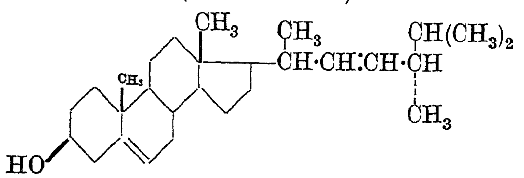
$C_{12}H_{10}O_5$ MW, 234

Needles from AcOEt. M.p. 254–5°.

Me ester: $C_{13}H_{12}O_5$. MW, 248. M.p. 155°.

Späth, Pesta, *Ber.*, 1933, **66**, 759.

Ostreasterol (*Chalinasterol ?*)



Probable structure

$C_{28}H_{46}O$ MW, 398

Occurs in oysters. M.p. 142–3°. $[\alpha]_D^{20}$ − 43·57° in $CHCl_3$.

Acetyl deriv.: m.p. 134·5°. $[\alpha]_D^{20}$ − 45·95° in $CHCl_3$.

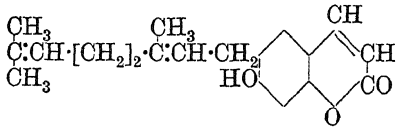
Propionyl: m.p. 113–14°.

Benzoyl: m.p. 145–7°.

Bergmann, *J. Biol. Chem.*, 1934, **104**, 317, 553.

Bergmann, Low, *J. Org. Chem.*, 1947, **12**, 67.

Ostruthin



$C_{19}H_{22}O_3$ MW, 298

Occurs in root of *Imperatoria ostruthium*, Linn. Cryst. from EtOH.Aq. M.p. 119° (117°). Sol. $CHCl_3$, AcOEt, hot EtOH. Insol. H_2O, pet. ether.

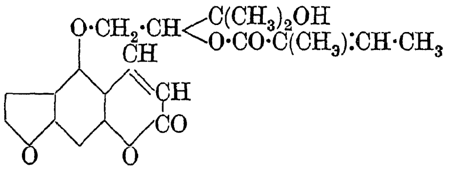
Acetyl deriv.: m.p. 80°.

Me ether: $C_{20}H_{24}O_3$. MW, 312. M.p. 55–55·5°.

Späth, Klager, *Ber.*, 1934, **67**, 859.

Butenandt, Marten, *Ann.*, 1932, **495**, 197.

Ostruthol



Probable structure

$C_{21}H_{22}O_7$ MW, 386

Occurs in *Imperatoria ostruthium*, Linn. Cryst. from C_6H_6. M.p. 136–7°. B.p. 225°/0·015 mm. $[\alpha]_D^{15}$ − 18·3° in Py.

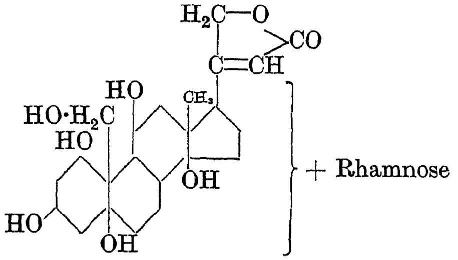
Acetyl deriv.: m.p. 125°.

Späth, Christiani, *Ber.*, 1933, **66**, 1150.

Osyritrin.

See Rutin.

Ouabain (*Acocantherin*, *g-strophanthin*)



Suggested structure

$C_{29}H_{44}O_{12}$ MW, 584

Glycoside occurring in *Acocanthera oabaio*. M.p. 180° (indefinite). Hyd. ⟶ rhamnose + acocanthic acid lactone.

Moir, *Chem. Abstracts*, 1924, **18**, 1825.

Fieser, Newman, *J. Biol. Chem.*, 1936, **114**, 705.

Tschesche, Haupt, *Ber.*, 1937, **70**, 43.

Chakravorty, Wallis, *J. Am. Chem. Soc.*, 1938, **60**, 1379.

Marker *et al.*, *J. Am. Chem. Soc.*, 1942, **64**, 720.

Mannich, Siewert, *Ber.*, 1942, **75**, 737, 750.

Ovoflavine.
See Lactoflavine.

Oxalacetic Acid (*Ketosuccinic acid, hydroxyfumaric acid, hydroxymaleic acid*)

HOOC·CO·CH_2·COOH
(2, 1)
I

HOOC·C(OH)
‖
HC·COOH
II

HOOC·C(OH)
‖
HOOC·CH
III

$C_4H_4O_5$ MW, 132

II.
Cryst. from Me_2CO–C_6H_6. M.p. 184°. Sol. H_2O, EtOH, Et_2O. Insol. $CHCl_3$, C_6H_6. Heat of comb. C_p 274·10 Cal., C_v 275·78 Cal. $k = 2{\cdot}76 \times 10^{-3}$ at 17°. Py at 50° ⟶ III.

III.
Cryst. from Me_2CO–C_6H_6. M.p. 152°. Sol. EtOH, Me_2CO, AcOEt. Spar. sol. Et_2O. Insol. C_6H_6, $CHCl_3$, ligroin. Heat of comb. C_p 284·9 Cal., C_v 286·58 Cal. $k = 2{\cdot}505 \times 10^{-3}$ at 17°.

I.
A,2NH_3 : m.p. 75–7° decomp.
A,$(CO(NH_2)_2)_2$: m.p. 124° decomp.
Di-brucine salt: m.p. 163–6°. Esters and salts are probably of I.
Di-Me ester: $C_6H_8O_5$. MW, 160. M.p. 74° (77°). *Me ether*: $C_7H_{10}O_5$. MW, 174. B.p. 228–30°.
1-*Et ester*: $C_6H_8O_5$. MW, 160. M.p. 102–3°. Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6.
Di-Et ester: $C_8H_{12}O_5$. MW, 188. B.p. 131–2°/24 mm. D_4^{20} 1·130–1·132. $n_D^{16\cdot6}$ 1·45614. *Semicarbazone*: m.p. 162°. p-*Chlorophenylhydrazone*: m.p. 119–20°. p-*Tolylhydrazone*: m.p. 105–6°.
1-*Me*: 2-*Et ester*: $C_7H_{10}O_5$. MW, 174. B.p. 130°/22 mm., 124°/16 mm. *Cu Salt*: green prisms from EtOH. M.p. 134–5°.
2-*Me*: 1-*Et ester*: b.p. 110°/13 mm. *Cu salt*: green needles. M.p. 165–6° (173–4° anhyd.).
2-*Et ester*: 1-*nitrile*: *oxime*: isonitrososuccinic acid Et ester nitrile. M.p. 104°. *Acetyl deriv.*: m.p. 146°.
Di-isoamyl ester: $C_{14}H_{24}O_5$. MW, 272. B.p. 167°/23 mm. *Cu salt*: green needles from EtOH. M.p. 83–5°.
Diamide: $C_4H_6O_3N_2$. MW, 130. M.p. 180° decomp.
Anhydride: *acetyl deriv.*: acetoxymaleic anhydride. M.p. 89–91°.
Oxime: isonitrososuccinic acid. (i) M.p. 126° decomp. (ii) M.p. 88° decomp.
2 : 4-*Dinitrophenylhydrazone*: m.p. 211° decomp.

Diels, Meyer, *Ann.*, 1934, **513**, 139.
U.S. Industrial Alcohol Co., U.S.P., 1,948,201, (*Chem. Abstracts*, 1934, **28**, 2730).
Blanchetière, *Compt. rend.*, 1916, **163**, 206.
Hantzsch, *Ber.*, 1915, **48**, 1407.
Gault, *Compt. rend.*, 1914, **158**, 711.
Fenton, Wilks, *J. Chem. Soc.*, 1912, **101**, 1570.
Meyer, *Ber.*, 1912, **45**, 2860.

Oxalacetoacetic Ethyl Ester

CO——CO
C_2H_5O·CO·CH·CO·CH_2
I

CO——C(OH)
C_2H_5O·CO·CH·CO·CH
II

CO——CH_2
C_2H_5O·CO·C:C(OH)·CO
III

$C_8H_8O_5$ MW, 184

(a) Yellow needles from Me_2CO. M.p. 145–50° decomp. Sol. H_2O, hot EtOH, hot AcOH. Spar. sol. Et_2O, ligroin.
Phenylhydrazone: m.p. 184–6° decomp.
Di-phenylhydrazone: m.p. 198–202°.
(b) Yellow leaflets from AcOEt. M.p. 105–10°. Sol. H_2O. Mod. sol. ord. org. solvents.

Wislicenus, Schollkopf, *J. prakt. Chem.*, 1917, **95**, 269.

Oxalan (*Oxaluramide*)

NH_2·CO·NH·CO·CO·NH_2

$C_3H_5O_3N_3$ MW, 131

Cryst. Does not melt below 310°. Insol. H_2O. Sol. H_2SO_4, repptd by H_2O.

Schenck, *Ber.*, 1905, **38**, 459.
Seemann, *Z. physik. Chem.*, 1905, **44**, 244.

Oxalic Acid

COOH
|
COOH

$C_2H_2O_4$ MW, 90

Occurs in many plants. Cryst. + $2H_2O$ from H_2O. M.p. 101·5°, anhyd. 189·5°. Dehydrates on heating and sublimes at 150–60°. Sol. 10·5 parts H_2O at 15°. Mod. sol. EtOH. Spar. sol. Et_2O. $Ca^{··}$ and $Hg^{·}$ salts very spar. sol. H_2O. $k_1 = 5{\cdot}36 \times 10^{-2}$, $k_2 = 5{\cdot}3 \times 10^{-5}$ at 25°. $D_4^{18\cdot5}$ 1·653. Heat of comb. C_p 60·2 Cal., C_v 61·1 Cal.
Me ester: $C_3H_4O_4$. MW, 104. B.p. 108–9°.
Di-Me ester: *see* Dimethyl oxalate.
Me-Et ester: $C_5H_8O_4$. MW, 132. B.p. 173·7°. D_0^0 1·5505.
Et ester: $C_4H_6O_4$. MW, 118. B.p. 117°/15 mm. D_4^{20} 1·2175. *Hydrazide*: m.p. 52–3° decomp.

Di-Et ester : *see* Diethyl oxalate.
Ethylene ester : $C_4H_4O_4$. MW, 116. M.p. 144°. Readily polymerises.
Propyl ester : $C_5H_8O_4$. MW, 132. B.p. 118–19°/13 mm.
Dipropyl ester : $C_8H_{14}O_4$. MW, 174. M.p. — 46·3°. B.p. 214–15° (211–12°). D_4^{20} 1·01693. n_D^{20} 1·4168.
Isopropyl ester : $C_5H_8O_4$. MW, 132. B.p. 111°/13 mm. D_4^{20} 1·1657.
Di-isopropyl ester : $C_8H_{14}O_4$. MW, 174. B.p. 189° (193–4°). D_4^{20} 1·00097. n_D^{20} 1·4100.
Dibutyl ester : $C_{10}H_{18}O_4$. MW, 202. F.p. — 29·6°. B.p. 247–9°. D_4^{20} 0·98732. n_D^{20} 1·4234.
Di-isobutyl ester : b.p. 229–31°. D_4^{20} 0·97373. n_D^{20} 1·4180.
*Di-*tert.*-butyl ester* : m.p. 71°.
*Di-*n*-amyl ester* : $C_{12}H_{22}O_4$. MW, 230. M.p. — 12·8°. B.p. 154·1°/14·8 mm. D^{20} 0·9722. n_D^{20} 1·4302.
Di-isoamyl ester : b.p. 267–8°.
*Di-*n*-hexyl ester* : $C_{14}H_{26}O_4$. MW, 258. M.p. — 9·0°. B.p. 135–6°/2·5 mm. D^{20} 0·9523. n_D^{20} 1·4331.
*Di-*n*-heptyl ester* : $C_{16}H_{30}O_4$. MW, 286. M.p. 12·5°. B.p. 142–4°/2 mm. D^{20} 0·9393. n_D^{20} 1·4372.
*Di-*n*-nonyl ester* : $C_{18}H_{34}O_4$. MW, 314. M.p. 11·2°. B.p. 167–9°/3 mm. D^{20} 0·9293. n_D^{20} 1·4404.
Dimyricyl ester : $C_{62}H_{122}O_4$. MW, 930. M.p. 91°.
Diallyl ester : $C_8H_{10}O_4$. MW, 170. B.p. 206–7°/754 mm.
Dicyclohexyl ester : $C_{14}H_{22}O_4$. MW, 254. M.p. 42°.
Diphenyl ester : $C_{14}H_{10}O_4$. MW, 242. M.p. 134°.
*Di-*o*-nitrophenyl ester* : $C_{14}H_8O_8N_2$. MW, 332. M.p. 185°.
*Di-*o*-tolyl ester* : $C_{16}H_{14}O_4$. MW, 270. M.p. 90–1°.
*Di-*m*-tolyl ester* : m.p. 105°.
*Di-*p*-tolyl ester* : m.p. 148°.
*Di-*p*-nitrobenzyl ester* : $C_{16}H_{12}O_8N_2$. MW, 360. M.p. 204°.
p*-Bromophenacyl ester* : fine white plates from Me_2CO. M.p. 242° decomp. Spar. sol. EtOH.
Me ester chloride : $C_3H_3O_3Cl$. MW, 122·5. B.p. 125° (118–20°). D_4^{20} 1·33163.
Et ester chloride : $C_4H_5O_3Cl$. MW, 136·5. B.p. 135°, 30°/10 mm. D_4^{20} 1·2226.
Propyl ester chloride : $C_5H_7O_3Cl$. MW, 150·5. B.p. 156–8°, 50°/12 mm. D_4^{20} 1·16697.
Isobutyl ester chloride : $C_6H_9O_3Cl$. MW, 164·5. B.p. 163–5°, 52°/10 mm. D_4^{20} 1·11532.
Isoamyl ester chloride : $C_7H_{11}O_3Cl$. MW, 178·5. B.p. 183–5°, 68°/10 mm. D_4^{20} 1·09312.
Dichloride : *see* Oxalyl chloride.
Monoamide : *see* Oxamic Acid.
Diamide : *see* Oxamide.
Methylamide : *see* Methyloxamic Acid.
sym.*-Dimethylamide* : *see sym.*-Dimethyloxamide.
unsym.*-Dimethylamide* : *see* Dimethyloxamic Acid.
Mononitrile : *see* Cyanoformic Acid.
Amide-nitrile : *see under* Cyanoformic Acid.
Dinitrile : *see* Cyanogen.
Monoanilide : *see* Oxanilic Acid.
Nitroanilide : *see* Nitro-oxanilic Acid.
Dianilide : *see* Oxanilide.
*Di-*o*-nitroanilide* : yellow plates from aniline. M.p. 331° part. decomp.
*Di-*m*-nitroanilide* : needles from aniline. M.p. 309–10°.
*Di-*p*-nitroanilide* : yellowish needles from aniline. M.p. 358–9° (260°).
Hydrazide : does not melt below 300°.
Dihydrazide : m.p. 241° decomp. N : N′*-Diacetyl* : m.p. 276°. *Hexa-acetyl* : m.p. 156–8°.

Johnson, Partington, *J. Chem. Soc.*, 1930, 1510.
Semenov, Shagalov, Astrakhantzev, *Chem. Abstracts*, 1935, **29**, 6882.
Chrzaszcz, Zakomorry, *Biochem. Z.*, 1935, **279**, 64.
Skinner, *J. Am. Chem. Soc.*, 1933, **55**, 2036.
Rakusin, *Chem.-Ztg.*, 1931, **55**, 128.
Jewel, Butts, *J. Am. Chem. Soc.*, 1931, **53**, 3560.
Sah, Chen, *ibid.*, 3901.
Mugdan, Sint, D.R.P., 606,774, (*Chem. Abstracts*, 1935, **29**, 3691).
du Pont, U.S.P., 1,948,441, (*Chem. Abstracts*, 1934, **28**, 2728).

2-Oxalobutyric Acid

HOOC·CO·CH(CH_3)·CH_2·COOH
I

HOOC·C(OH):C(CH_3)·CH_2·COOH
II

$C_6H_8O_5$ MW, 160

Di-Et ester : $C_{10}H_{16}O_5$. MW, 216. B.p. 163°/5 mm.

Feist, Brewer, *Ann.*, 1922, **428**, 68.

Oxalocrotonic Acid (1-*Hydroxymuconic acid*)

HOOC·CO·CH_2·CH:CH·COOH
I

HOOC·C(OH):CH·CH:CH·COOH
II

$C_6H_6O_5$ MW, 158

Yellow cryst. M.p. about 190° decomp. Sol. EtOH, AcOH, H·COOH. Spar. sol. H_2O. Insol. C_6H_6, $CHCl_3$.

Lapworth, *J. Chem. Soc.*, 1901, **79**, 1279.

Oxalodiacetic Acid.
See Ketipic Acid.

Oxalomalonic Acid

$$\mathrm{HOOC{\cdot}CO{\cdot}CH}<\begin{matrix}\mathrm{COOH}\\ \mathrm{COOH}\end{matrix}$$

I

$$\mathrm{HOOC{\cdot}C(OH){:}C}<\begin{matrix}\mathrm{COOH}\\ \mathrm{COOH}\end{matrix}$$

II

$C_5H_4O_7$ MW, 176

Tri-Me ester: $C_8H_{10}O_7$. MW, 218. Needles from Et_2O. M.p. 49–50°. Sol. Et_2O, C_6H_6, $CHCl_3$. Spar. sol. EtOH.

Tri-Et ester: $C_{11}H_{16}O_7$. MW, 260. B.p. 220°/10 mm. D_4^{20} 1·153. n_D^{20} 1·4468.

Auwers, Auffenberg, *Ber.*, 1918, **51**, 1103.

Oxalomethylaniline.
See N-Methyloxanilic Acid.

1-Oxalopropionic Acid (*Methyloxalacetic acid*)

$$\begin{matrix}\mathrm{CH_3}\\ |\\ \mathrm{HOOC{\cdot}CH{\cdot}CO{\cdot}COOH}\end{matrix}$$

$C_5H_6O_5$ MW, 146

Di-Et ester: $C_9H_{14}O_5$. MW, 202. Liq. B.p. 137–8°/23 mm., 114–16°/10 mm. Misc. with EtOH, Et_2O. Insol. H_2O. Decomp. on standing. Alc. $FeCl_3$ ⟶ intense red col.

Monoamide: $C_5H_7O_4N$. MW, 145. Not isolated. *Phenylhydrazone*: m.p. 99–100°.

Mononitrile: $C_5H_5O_3N$. MW, 127. Yellow cryst. from Et_2O-pet. ether. M.p. 207–8°. Sol. H_2O. Insol. C_6H_6. Reacts acid. Alc. $FeCl_3$ ⟶ red col.

Wislicenus, Arnold, *Ann.*, 1888, **246**, 329.
Wislicenus, Silberstein, *Ber.*, 1910, **43**, 1829.

Oxalosuccinic Acid

$$\begin{matrix}\mathrm{HOOC{\cdot}CO{\cdot}CH{\cdot}COOH}\\ \mathrm{CH_2{\cdot}COOH}\end{matrix}$$

I

$$\begin{matrix}\mathrm{HOOC{\cdot}C(OH){:}C{\cdot}COOH}\\ \mathrm{CH_2{\cdot}COOH}\end{matrix}$$

II

$C_6H_6O_7$ MW, 190

Tri-Et ester: $C_{12}H_{18}O_7$. MW, 274. B.p. 170–5°/12–13 mm. *Phenylhydrazone*: decomp. at 85°.

Wislicenus, Waldmüller, *Ber.*, 1911, **44**, 1564.
Friedman, Kosower, *Organic Syntheses*, 1946, XXVI, 42.

Oxaluramide.
See Oxalan.

Oxaluric Acid (*Mono-oxalylurea*)

$$\mathrm{HOOC{\cdot}CO{\cdot}NH{\cdot}CO{\cdot}NH_2}$$

$C_3H_4O_4N_2$ MW, 132

Cryst. Decomp. at 208–10°. Sol. H_2O. Spar. sol. ord. org. solvents. Heat of comb. 207·7 Cal. Hot H_2O ⟶ oxalic acid + urea.

Me ester: $C_4H_6O_4N_2$. MW, 146. M.p. 192° decomp.

Et ester: $C_5H_8O_4N_2$. MW, 160. M.p. 177–8° decomp.

ω-N-*Formyl*: m.p. 175° decomp.

Amide: *see* Oxalan.

Hydrazide: m.p. 198° decomp.

Fosse, Thomas, Graeve, *Compt. rend.*, 1935, **200**, 1260.
Biltz, Schauder, *J. prakt. Chem.*, 1923, **106**, 147.

Oxalyl bromide

$$\begin{matrix}\mathrm{COBr}\\ |\\ \mathrm{COBr}\end{matrix}$$

$C_2O_2Br_2$ MW, 216

Green liq. M.p. — 19·5°. B.p. 102–3°/720 mm., 16–17°/10 mm.

Staudinger, Anthes, *Ber.*, 1913, **46**, 1431.

Oxalylcarbanilide.
See Diphenylparabanic Acid.

Oxalyl chloride

$$\begin{matrix}\mathrm{COCl}\\ |\\ \mathrm{COCl}\end{matrix}$$

$C_2O_2Cl_2$ MW, 127

Needles from Et_2O or pet. ether at — 80°. M.p. — 12°. B.p. 63·5–64°/763 mm. $D_4^{13\cdot4}$ 1·4884. $n_D^{12\cdot8}$ 1·434. Forms add. comp., m.p. 68°, with dioxan in pet. ether at — 5°.

Staudinger, *Ber.*, 1908, **41**, 3563; D.R.Ps., 216,918–19, (*Chem. Abstracts*, 1910, **4**, 1087).
Jones, Tasker, *Proc. Chem. Soc.*, 1908, **24**, 271.
Giua, *Chem. Abstracts*, 1925, **19**, 1245.
Varvoglis, *Ber.*, 1938, **71**, 32.

Oxalyldiacetic Acid.
See Ketipic Acid.

Oxalyl-diacetophenone.
See Diphenacyl Diketone.

Oxalyldimethylurea.
See Dimethylparabanic Acid.

Oxalyl fluoride

$$\begin{matrix}\mathrm{COF}\\ |\\ \mathrm{COF}\end{matrix}$$

$C_2O_2F_2$ MW, 94

Colourless liq. B.p. 26°.

Fukuhara, Bigelow, *J. Am. Chem. Soc.*, 1941, **63**, 788.

Oxalylguanidine

$$\begin{matrix}\mathrm{CO{\cdot}NH}\\ |\\ \mathrm{CO{\cdot}NH}\end{matrix}>\mathrm{C{:}NH}$$

$C_3H_3O_2N_3$ MW, 113

Prisms from H_2O. M.p. 266–8° decomp. (sealed tube). Sol. H_2O ⟶ oxalic acid + guanidine slowly. Insol. EtOH.

Traube, *Ber.*, 1893, **26**, 2552.

Oxalylmethylurea.
See Methylparabanic Acid.

Oxalylurea.
See Oxaluric Acid *and* Parabanic Acid.

Oxamethane.

See under Oxamic Acid.

Oxamic Acid (*Oxaminic acid, oxalic acid monoamide*)

$CONH_2$
$COOH$

$C_2H_3O_3N$ MW, 89

Cryst. from H_2O. M.p. 210° decomp. Spar. sol. H_2O. Insol. EtOH, Et_2O. Heat of comb. C_p 132·0 Cal., C_v 129·5 Cal.

Me ester : $C_3H_5O_3N$. MW, 103. M.p. 122–3°.

Et ester : oxamethane. $C_4H_7O_3N$. MW, 117. M.p. 114–15°. N-*Acetyl* : m.p. 53·5–54·5°.

Pentachloroethyl ester : $C_4H_2O_3NCl_5$. MW, 289·5. M.p. 134°.

Propyl ester : $C_5H_9O_3N$. MW, 131. M.p. 90–2°.

Isopropyl ester : m.p. 86–7°.

Butyl ester : $C_6H_{11}O_3N$. MW, 145. M.p. 82–4°.

Isobutyl ester : m.p. 89–90°.

Isoamyl ester : $C_7H_{13}O_3N$. MW, 159. M.p. 92–3°.

Amide : *see* Oxamide.

Hydrazide : semioxamazide. M.p. 220–1° decomp. (223–4°).

Diacetyl : $H_2N·CO·CO·NH·N(CO·CH_3)_2$. M.p. 184–5°.

N-*Me* : *see* Methyloxamic Acid.

N-*Di-Me* : *see* Dimethyloxamic Acid.

Nitroanilide : *see under* Nitro-oxanilic Acid.

Sah, Chen, *J. Am. Chem. Soc.*, 1931, **53**, 3901.
Oelkers, *Ber.*, 1889, **22**, 1569.
Weddige, *J. prakt. Chem.*, 1874, **10**, 196.

Oxamide (*Oxalic acid diamide*)

$CONH_2$
$CONH_2$

$C_2H_4O_2N_2$ MW, 88

Needles. Spar. sol. EtOH, hot H_2O. Decomp. above 320°. Heat of comb. C_p 203·3 Cal.

N : N′-*Dipropionyl* : m.p. 216° decomp.
N : N′-*Dibutyryl* : m.p. 197°.
N : N′-*Di-isobutyryl* : m.p. 160°.
N : N′-*Dibenzyl* : m.p. 222°.

Kasiwagi, *Bull. Chem. Soc. Japan*, 1926, **1**, 67.
Langebeck, *Ann.*, 1929, **469**, 16.
Bucher, Can. P. 173,369, (*Chem. Abstracts*, 1918, **12**, 156); U.S.P., 1,194,354, (*Chem. Abstracts*, 1916, **10**, 2500).

Oxanilic Acid (*Oxalic acid monoanilide*)

$COOH$
$CO·NH·C_6H_5$

$C_8H_7O_3N$ MW, 165

Needles from C_6H_6. M.p. 148–9° (149–50°). Sol. EtOH, Et_2O, $CHCl_3$. Mod. sol. hot H_2O. Spar. sol. C_6H_6, ligroin. $k = 1·21 \times 10^{-2}$ at 25°. Heat of comb. C_p 863·1 Cal.

Me ester : $C_9H_9O_3N$. MW, 179. M.p. 114° (111–12°).

Et ester : $C_{10}H_{11}O_3N$. MW, 193. M.p. 66–7°. Heat of comb. C_p 1191·2 Cal. N-*Acetyl* : m.p. 64–5°.

Propyl ester : $C_{11}H_{13}O_3N$. MW, 207. M.p. 92°.

Isopropyl ester : m.p. 52°.

Isobutyl ester : $C_{12}H_{15}O_3N$. MW, 221. M.p. 85°.

Isoamyl ester : $C_{13}H_{17}O_3N$. MW, 235. M.p. 50°.

m-*Tolyl ester* : $C_{15}H_{13}O_3N$. MW, 255. M.p. 94°.

p-*Tolyl ester* : m.p. 132°.

Chloride : $C_8H_6O_2NCl$. MW, 183·5. M.p. 82·5°.

Amide : $C_8H_8O_2N_2$. MW, 164. M.p. 228° (224–5°).

Nitrile : $C_8H_6ON_2$. MW, 146. M.p. 128° decomp.

Anilide : *see* Oxanilide.

Aschan, *Ber.*, 1890, **23**, 1820.
Stobbe, Knebel, *Ber.*, 1921, **54**, 1216.

Oxanilide (sym.-*Diphenyloxamide, oxalic dianilide*)

$CO·NH·C_6H_5$
$CO·NH·C_6H_5$

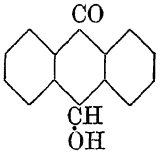
$C_{14}H_{12}O_2N_2$ MW, 240

Leaflets from C_6H_6 or $PhNO_2$. M.p. 254° (252–3°). B.p. above 360°. Sol. C_6H_6. Spar. sol. hot EtOH. Insol. H_2O, Et_2O. Heat of comb. C_p 1665·4 Cal. Forms a *N*-Na deriv.

N-*Acetyl* : m.p. 197–8°.
N : N′-*Diacetyl* : m.p. 208–9° decomp.
N : N′-*Dibutyryl* : m.p. 156°.
N : N′-*Dibenzoyl* : m.p. 212–13° decomp. (210°).

Bornwater, *Rec. trav. chim.*, 1912, **31**, 108.
Macullum, *J. Soc. Chem. Ind.*, 1923, **42**, 468T.

Oxanthranol (10-*Hydroxyanthrone, oxanthrone*)



$C_{14}H_{10}O_2$ MW, 210

Keto form of anthrahydroquinone. Colourless cryst. M.p. 167° decomp. Sols. do not fluoresce. Stable in air. Insol. cold alkalis. Sol. hot alc. alkalis, isomerising to anthrahydroquinone. Zn + AcOH ⟶ anthranol.

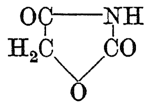
Acetyl : m.p. 108–9°.

Me ether : 10-methoxyanthrone. $C_{15}H_{12}O_2$. MW, 224. M.p. 102·5° (98°).

Meyer, *Ann.*, 1911, **379**, 60, 77.

Oxanthrone.
See Oxanthranol.

2 : 4-Oxazolidinedione

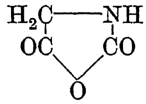


$C_3H_3O_3N$ MW, 101

Cryst. M.p. 89–90°. B.p. 173°/11 mm. Sol. H_2O giving acid sol.

Wallingford, Thorpe, Stoughton, *J. Am. Chem. Soc.*, 1945, **67**, 522.

2 : 5-Oxazolidinedione (*Anhydrocarboxyglycine*)



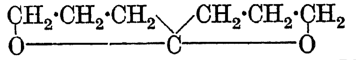
$C_3H_3O_3N$ MW, 101

Cryst. M.p. 100°. On standing loses CO_2 to give a high molecular weight compound.

Go, Tani, *Bull. Chem. Soc. Japan*, 1939, **14**, 510, (*Chem. Abstracts*, 1940, **34**, 1971).

Oxeserolene.
See Hydroxyeserolene.

Oxetone



$C_7H_{12}O_2$ MW, 128

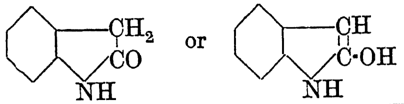
F.p. below — 17°. B.p. 159·4°. Mod. sol. cold H_2O. Spar. sol. hot H_2O. Spar. sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Reduces Tollen's reagent.

Fittig, Ström, *Ann.*, 1892, **267**, 197.
Granichstädten, Werner, *Monatsh.*, 1901, 22, 333.

Oximino-.
See also Isonitroso-.

Oximinoadipic Acid.
See under 1-Ketoadipic Acid.

Oxindole (*2-Hydroxyindole*)



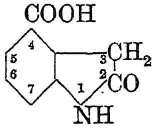
C_8H_7ON MW, 133

Needles from H_2O. M.p. 127°. B.p. 227°/23 mm. Sol. EtOH, Et_2O, hot H_2O. Reduces Tollen's reagent.

N-*Acetyl*: m.p. 126°.

Stollé, *J. prakt. Chem.*, 1930, **128**, 1
Heller, *Ber.*, 1916, **49**, 2775.
Marschalk, *J. prakt. Chem.*, 1914, **88**, 234.
Di Carlo, *J. Am. Chem. Soc.*, 1944, **66**, 1420.
Sumpter, *Chem. Reviews*, 1945, **37**, 443.

Oxindole-4-carboxylic Acid



$C_9H_7O_3N$ MW, 177

Yellow cryst. Decomp. above 280°.

Braun, Hahn, *Ber.*, 1923, **56**, 2345.

Oxindole-6-carboxylic Acid.

Brownish-yellow cryst. from EtOH.Aq. M.p. 313°. Spar. sol. ord. org. solvents.

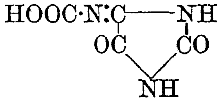
Fileti, Cairola, *Gazz. chim. ital.*, 1892, **22**, ii, 392.

Oxindone.
See 1 : 2-Diketohydrindene.

Oxine.
See 8-Hydroxyquinoline.

Oxisatin.
See Coumarandione.

Oxonic Acid (*Allantoxanic acid*)



$C_4H_3O_4N_3$ MW, 157

M.p. anhyd. about 261° decomp.

Biltz, Robi, *Ber.*, 1920, **53**, 1967.

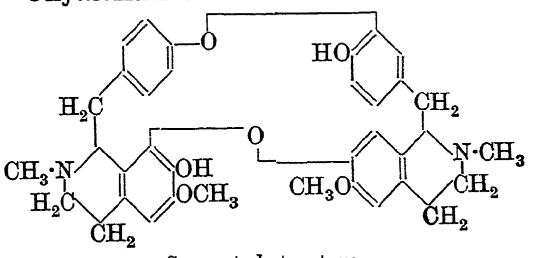
Oxonitine

$C_{31}H_{41}O_{12}N$ MW, 619

Needles from AcOH–Me_2CO. M.p. 277° decomp. Spar. sol. ord. org. solvents. $[\alpha]_D^{16}$ — 48·18° in $CHCl_3$.

Majima, Suginome, Shimanuki, *Ber.*, 1932, **65**, 595 (*Bibl.*).
Lawson, *J. Chem. Soc.*, 1936, 82.
Suginome, *Ann.*, 1937, **533**, 172; *Chem. Abstracts*, 1938, **32**, 6654.
Jacobs, Elderfield, Craig, *J. Biol. Chem.*, 1939, **128**, 439.
Jacobs, Craig, *J. Biol. Chem.*, 1940, **136**, 323.

Oxyacanthine



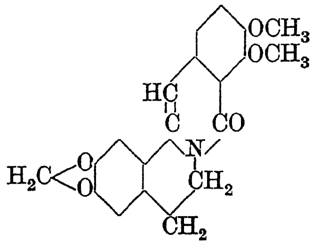
Suggested structure

$C_{38}H_{38}O_6N_2$ MW, 618

Occurs in root of *Berberis vulgaris*. Needles from EtOH. M.p. 217° (208–14°). Sol. $CHCl_3$, C_6H_6. Insol. ligroin. $[\alpha]_D + 131{\cdot}6°$ in $CHCl_3$.
B,HCl: m.p. 270°.
B,HBr: m.p. 273°.
Me ether: $C_{39}H_{40}O_6N_2$. MW, 632. *B,HCl*: m.p. 261°.

Bruchhausen, Gericke, *Chem. Zentr.*, 1931, I, 2761.
Kondo, Tomita, *Chem. Abstracts*, 1939, **33**, 627.

Oxyberberine



$C_{20}H_{17}O_5N$ MW, 351

Yellow needles from EtOH. M.p. 198–200°. Sol. hot AcOH. Spar. sol. EtOH, C_6H_6. Insol. ligroin.

Perkin, Ray, Robinson, *J. Chem. Soc.*, 1925, 742.
Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 552.
Späth, Quietensky, *Ber.*, 1925, **58**, 2267.

Oxycyanogen (*Oxycyan*)

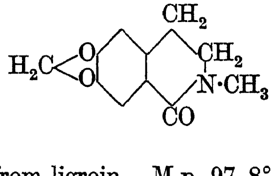
$(CNO)_x$

$(CNO)_x$ MW, $(42)_x$

Plates. M.p. — 12·5 to — 11·5°. Decomp. at ord. temp. except in solution. Sol. CS_2, CCl_4. Liberates I from KI.

Hunt, *J. Am. Chem. Soc.*, 1932, **54**, 907.

Oxyhydrastinine



$C_{11}H_{11}O_3N$ MW, 205

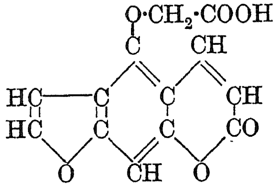
Needles from ligroin. M.p. 97–8°. Mod. sol. EtOH, $CHCl_3$, C_6H_6, AcOEt, CS_2. $KMnO_4 \longrightarrow$ hydrastic acid.
B,HBr: m.p. 200°.
B_2,H_2PtCl_6: m.p. 160°.

Freund, *Ber.*, 1889, **22**, 457.
Perkin, *J. Chem. Soc.*, 1890, **57**, 1034.

Oxy-Koch Acid.
See 1-Naphthol-3 : 6 : 8-trisulphonic Acid.

Oxyneurine.
See Betaine.

Oxypeucedanic Acid

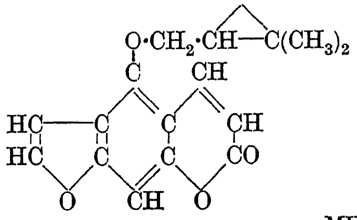


$C_{13}H_8O_6$ MW, 260

Cryst. from AcOH.Aq. M.p. 265°.
Me ester: $C_{14}H_{10}O_6$. MW, 274. Cryst. from MeOH.Aq. M.p. 185°.

Späth, Klager, *Ber.*, 1933, **66**, 921.
Butenandt, Marten, *Ann.*, 1932, **495**, 209.

Oxypeucedanine

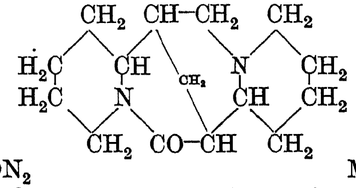


$C_{16}H_{14}O_5$ MW, 286

Occurs in root of *Imperatoria ostruthium*, Linn. Cryst. from Et_2O–$CHCl_3$. M.p. 142–3°. KOH fusion $\longrightarrow$ phloroglucinol.
Hydrate: cryst. from AcOEt or MeOH.Aq. M.p. 134°. *Monoacetyl*: cryst. from Me_2CO–pet. ether. M.p. 139°. *Diacetyl*: cryst. from $CHCl_3$–pet. ether. M.p. 132–3° (136°). *Monobenzoyl*: cryst. from EtOH.Aq. M.p. 172–172·5°. *Phenylurethane*: cryst. from AcOEt–pet. ether. M.p. 174°.

Späth, Klager, *Ber.*, 1933, **66**, 914.
Butenandt, Marten, *Ann.*, 1932, **495**, 205.
Späth, Dobrovolny, *Ber.*, 1939, **72**, 52.

Oxysparteine (*Isolupanine*)



$C_{15}H_{24}ON_2$ MW, 248

Cryst. from pet. ether. M.p. 111°.
B,HI: prisms from EtOH. M.p. 275°.
Methiodide: m.p. 203–4°.

Clemo, Morgan, Raper, *J. Chem. Soc.*, 1936, 1025.

Oxytiglic Acid.
See 1 : 2-Dimethylglycidic Acid.

Ozobenzene (*Benzene triozonide*)

$C_6H_6O_9$ MW, 222

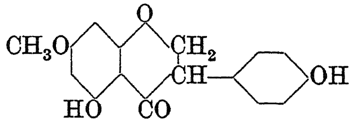
Amorp. Explodes at 50°. Very violent explosive. Insol. EtOH, Et_2O, $CHCl_3$, CS_2, ligroin. $H_2O \longrightarrow CO_2 + H{\cdot}COOH + CH_3{\cdot}COOH$.

Renard, *Bull. soc. chim.*, 1895, **13**, 940.
Anon., *Chem. Abstracts*, 1920, **14**, 1220.

P

Pachycarpine.
See Sparteine.

Padmakasein



$C_{16}H_{14}O_5$ MW, 286

Constituent of bark of *Prunus puddum*. Plates and prisms from EtOH. M.p. 236–8°. Conc. HNO_3 ⟶ green ⟶ red col. $FeCl_3$ ⟶ reddish-violet col.

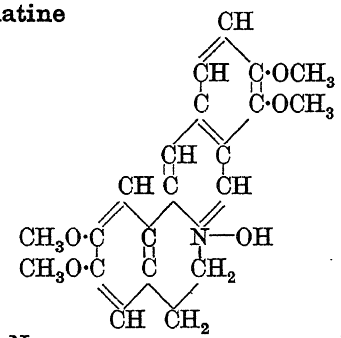
Mono-Me ether: $C_{17}H_{16}O_5$. MW, 300. Prisms from EtOH. M.p. 131–2°.

Di-Me ether: $C_{18}H_{16}O_5$. MW, 314. Cryst. from EtOH. M.p. 146–7°.

Narasimhachari, Seshadri, *Proc. Indian Acad. Sci.*, 1952, **35**, 202.

Pæonol.
See Peonol.

Palmatine



$C_{21}H_{23}O_5N$ MW, 369

Occurs in East African Calumba root.

Iodide: $C_{21}H_{22}O_4NI,2H_2O$. Orange-yellow needles from H_2O. M.p. 241° decomp. (238–40° decomp.). Mod. sol. hot H_2O, EtOH.

B,HNO₃,2H₂O: yellow needles. M.p. 239° decomp.

Chloride: $C_{21}H_{22}O_4NCl,3H_2O$. Greenish-yellow needles from H_2O. M.p. 205° decomp. Sol. H_2O, EtOH.

Sulphate: $(C_{21}H_{22}O_4N)_2SO_4,5H_2O$. Yellow needles. M.p. 250°. Sol. EtOH. Mod. sol. cold H_2O.

$(C_{21}H_{22}O_4NCl)_2,PtCl_4$: yellow cryst. M.p. 236°.

Feist, Sandstede, *Arch. Pharm.*, 1918, **256**, 1.
Feist, Awe, Etzrodt, *Chem. Zentr.*, 1935, I, 2374.
Späth, Quietensky, *Ber.*, 1925, **58**, 2267.
Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 548.
Späth, Meinhard, *Ber.*, 1942, **75**, 400.

Palmitaldehyde.
See Palmitic Aldehyde.

Palmitic Acid (*Hexadecylic acid*)

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH_2{\cdot}COOH$

$C_{16}H_{32}O_2$ MW, 256

Occurs in form of esters (glycerides) in oils and fats of vegetable and animal origin. Usually obtained from palm-oil, Japan wax or Chinese vegetable tallow. M.p. 63–4° (62·6°). B.p. 390°, 268·5°/100 mm., 219°/20 mm., 215°/15 mm. D_4^{62} 0·8527 (liq.), $D_4^{75\cdot8}$ 0·8465, D_4^{70} 0·8487, D_4^{90} 0·8347. n_D^{60} 1·4339, n_D^{70} 1·4304, $n_D^{74\cdot5}$ 1·4284, n_D^{80} 1·42691. Insol. H_2O. Sol. 11 parts EtOH at 20°, 100 parts 94–5% EtOH at 0°, 256 parts 75% EtOH at 20°, 2000 parts 50% EtOH at 10°. Sol. Et_2O. Spar. sol. pet. ether. Alk. $KMnO_4$ ⟶ mixture of mono- and di-carboxylic acids of smaller carbon content. Chars with conc. H_2SO_4 at 160–80° giving CO and SO_2. P_2O_5 at 200–10° ⟶ palmitone.

$C_{16}H_{31}O_2NH_4 + C_{16}H_{32}O_2$: m.p. 89°. Insol. cold H_2O.

$C_{16}H_{31}O_2NH_3CH_2{\cdot}C_6H_5$: m.p. 79–79·5°.

$C_{16}H_{31}O_2Na$: cryst. from EtOH. M.p. about 270°.

$2(C_{16}H_{31}O_2Na) + C_{16}H_{32}O_2$: m.p. 115–17°. Insol. H_2O. Sol. hot EtOH.

$C_{16}H_{31}O_2Na + C_{16}H_{32}O_2$: m.p. 97–8°.

$C_{16}H_{31}O_2Na + 2C_{16}H_{32}O_2$: m.p. 81–2°.

$C_{16}H_{31}O_2K + C_{16}H_{32}O_2$: cryst.

$C_{16}H_{31}O_2K$: white tablets. Sol. EtOH.

$(C_{16}H_{31}O_2)_2Ca$: spar. sol. H_2O.

Me ester: $C_{17}H_{34}O_2$. MW, 270. Cryst. from EtOH at −15°. M.p. 30·5° (29·5°). B.p. 415–18°/747 mm., 190·5° (196°)/15 mm., 184°/12 mm.

Et ester: $C_{18}H_{36}O_2$. MW, 284. Needles. α-Form, m.p. 24°. β-Form, m.p. 19·3°. B.p. 184·5–185·5° (191°)/10 mm. n_D^{50} 1·4278, n_D^{70} 1·4200.

2-*Chloroethyl ester*: $C_{18}H_{35}O_2Cl$. MW, 318·5. Plates from EtOH. M.p. 41·5° (about 44°). B.p. 138° in high vacuum.

2-*Bromoethyl ester*: $C_{18}H_{35}O_2Br$. MW, 363. M.p. 62°. B.p. 144° in high vacuum.

2-*Iodoethyl ester*: $C_{18}H_{35}O_2I$. MW, 410. Plates from EtOH. M.p. 54°.

Propyl ester: $C_{19}H_{38}O_2$. MW, 298. Needles. M.p. 20·4° (18·8–19·2°). n_D^{50} 1·4290, n_D^{70} 1·4211.

2 : 3-*Dichloropropyl ester*: m.p. 17°.

2 : 3-*Dibromopropyl ester*: m.p. 26°.

Butyl ester: $C_{20}H_{40}O_2$. MW, 312. M.p. 16·9°. n_D^{50} 1·4312, n_D^{70} 1·4232.

n-*Amyl ester*: $C_{21}H_{42}O_2$. MW, 326. M.p. 19·4°. n_D^{50} 1·4320, n_D^{70} 1·4241.

Isoamyl ester: m.p. 12·5° (9°). n_D^{50} 1·4315, n_D^{70} 1·4235.
Heptyl ester: $C_{23}H_{46}O_2$. MW, 354. Liq. Solidifies at 8–10°. B.p. 205°/3 mm. D_{15}^{15} 0·8642. n_D^{20} 1·4481.
Octyl ester: $C_{24}H_{48}O_2$. MW, 368. M.p. 22·5°. n_D^{50} 1·4358, n_D^{70} 1·4277.
Decyl ester: $C_{26}H_{52}O_2$. MW, 396. M.p. 30°.
Dodecyl ester: $C_{28}H_{56}O_2$. MW, 424. Plates from EtOH. M.p. 41°.
Tetradecyl ester: $C_{30}H_{60}O_2$. MW, 452. M.p. 48°.
Pentadecyl ester: $C_{31}H_{62}O_2$. MW, 466. M.p. 57° (55·5°).
Hexadecyl (cetyl) ester: $C_{32}H_{64}O_2$. MW, 480. Chief constituent of spermaceti. Plates from Et_2O. M.p. 53–4° (51·6°).
Octadecyl ester: $C_{34}H_{68}O_2$. MW, 508. M.p. 59°.
Ceryl ester: $C_{42}H_{84}O_2$. MW, 620. Main constituent of opium wax. M.p. 79°.
Melissyl ester: $C_{46}H_{92}O_2$. MW, 676. A constituent of beeswax. M.p. 72°.
Vinyl ester: $C_{18}H_{34}O_2$. MW, 282. B.p. 165°/2 mm.
Allyl ester: $C_{19}H_{36}O_2$. MW, 296. M.p. 20–5°.
2-*Chloroallyl ester*: m.p. 28–9°. B.p. 190–1°/4 mm. D_{41}^{30} 0·9291. n_D^{30} 1·4524.
Crotyl ester: b.p. 188–9°/3·8 mm. D_{41}^{30} 0·8616. n_D^{30} 1·4466.
Ethylene glycol mono-ester: $C_{18}H_{36}O_3$. MW, 300. M.p. 51·5°.
Ethylene glycol di-ester: $C_{34}H_{66}O_4$. MW, 538. M.p. 72° (69°). B.p. 226° in high vacuum.
Glycerol mono-ester: *see* Monopalmitin.
Glycerol di-ester: *see* Dipalmitin.
Glycerol tri-ester: *see* Tripalmitin.
Acetonyl ester: m.p. 50·5°. *Oxime*: m.p. 55·5–56°.
Phenyl ester: $C_{22}H_{36}O_2$. MW, 332. M.p. 45°. B.p. 249·5°/15 mm.
o-*Nitrophenyl ester*: $C_{22}H_{35}O_4N$. MW, 377. Needles from EtOH. M.p. 51–2°.
p-*Tolyl ester*: $C_{23}H_{38}O_2$. MW, 346. M.p. 47°. B.p. 258°/15 mm.
o-*Phenylene di-ester*: m.p. 58°.
m-*Phenylene di-ester*: m.p. 62·5–63·5°.
Benzyl ester: $C_{23}H_{38}O_2$. MW, 346. M.p. 36°.
p-*Nitrobenzyl ester*: $C_{23}H_{37}O_4N$. MW, 391. M.p. 42–42·5°.
Phenacyl ester: $C_{24}H_{38}O_3$. MW, 374. M.p. 63°.
p-*Chlorophenacyl ester*: $C_{24}H_{37}O_3Cl$. MW, 408·5. M.p. 82°.
p-*Bromophenacyl ester*: $C_{24}H_{37}O_3Br$. MW, 454. M.p. 86° (81·5°).
p-*Iodophenacyl ester*: $C_{24}H_{37}O_3I$. MW, 500. M.p. 90°.
p-*Nitrophenacyl ester*: $C_{24}H_{37}O_5N$. MW, 419. M.p. 42·5°.
p-*Phenylphenacyl ester*: m.p. 94°.
Furfuryl ester: b.p. 205–6°/3·8 mm. D_4^{30} 0·9226. n_D^{30} 1·4850.
Amide: $C_{16}H_{33}ON$. MW, 255. M.p. 106–7°. B.p. 235–6°/12 mm.
Anilide: needles from EtOH. M.p. 90·5° (87·5°). B.p. 282–4°/17 mm., 132·5°/10 mm. Sol. EtOH, Me_2CO, warm AcOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. pet. ether. Insol. H_2O.
p-*Bromoanilide*: m.p. 110°.
2 : 4 : 6-*Tribromoanilide*: m.p. 124°.
p-*Nitroanilide*: m.p. 93°.
o-*Toluidide*: m.p. 90–1°.
m-*Toluidide*: m.p. 74·5–75·5°.
p-*Toluidide*: cryst. from EtOH. M.p. 96° (93·5°). B.p. 140°/10 mm.
o-*Nitro*-p-*toluidide*: m.p. 78–9°.
1-*Naphthylamide*: needles. M.p. 112·8° (106°). B.p. 182°/10 mm.
2-*Naphthylamide*: cryst. from EtOH. M.p. 109°. B.p. 198·5°/10 mm.
Benzylamide: m.p. 94·5–95°.
Cyclohexylamide: m.p. 94–5°.
p-*Toluenesulphonamide*: m.p. 93–4°.
Saccharin amide: m.p. 90°.
Monoureide: m.p. 135–6°.
Phenylhydrazide: m.p. 110·5°.
2 : 4-*Dinitrophenylhydrazide*: m.p. 120–1°.
Diphenylhydrazide: m.p. 124°.
2-*Naphthylhydrazide*: m.p. 135°.
Anhydride: $C_{32}H_{62}O_3$. MW, 494. M.p. 64°. D_4^{70} 0·847, D_4^{83} 0·8383. n_D^{83} 1·4364, n_D^{100} 1·4679.
Chloride: $C_{16}H_{31}OCl$. MW, 274·5. M.p. 12°. B.p. 194–5°/17 mm., 198–200°/15 mm.
Nitrile: $C_{16}H_{31}N$. MW, 237. M.p. 31° (29°, 33°). B.p. 251°/100 mm., 196°/15 mm., 176–82°/6 mm., 167·4°/1 mm. D_4^{31} 0·8224 (liq.), D^{40} 0·8186.

Krafft, *Ber.*, 1888, **21**, 2265.
Chittenden, Smith, *Am. Chem. J.*, 1884, **6**, 218.
Claus, v. Dreden, *J. prakt. Chem.*, 1891, **43**, 149.
Klages, *Ber.*, 1902, **35**, 2260.
Kreis, Hafner, *Ber.*, 1903, **36**, 2769.
Charitschkow, *Chem. Zentr.*, 1905, II, 118.
Bückel, D.R.P., 281,364, (*Chem. Zentr.*, 1915, I, 230).
Holde, Ripper, Zadek, *Ber.*, 1924, **57**, 103.
Whitby, *J. Chem. Soc.*, 1926, 1462.
Wilkie, *J. Soc. Chem. Ind.*, 1927, **46**, 471T.
Hann, Reid, Jamieson, *J. Am. Chem. Soc.*, 1930, **52**, 818.
Dubovitz, *Chem.-Ztg.*, 1930, **54**, 814.
Smith, *J. Chem. Soc.*, 1931, 802.
Deutsche Hydrierwerke, A-G., D.R.P., 572,867, (*Chem. Abstracts*, 1933, **27**, 4433).

Palmitic Aldehyde (*Palmitaldehyde, hexadecylic aldehyde, hexadecanal*)

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH_2{\cdot}CHO$

$C_{16}H_{32}O$ MW, 240

M.p. 34°. B.p. 200–2°/29 mm. Sol. most org. solvents. Insol. H_2O. Polymerises on keeping to trimeride (needles from Et_2O, m.p. 73°).

Oxime: needles from dil. EtOH. M.p. 88°. Sol. Et_2O, $CHCl_3$. Spar. sol. pet. ether, C_6H_6.

Semicarbazone: plates from dil. EtOH. M.p. 109° (107°). Sol. hot $CHCl_3$, C_6H_6.

Thiosemicarbazone: m.p. 109°.

p-*Nitrophenylhydrazone*: yellow cryst. M.p. 96·5°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 105–7° corr.

Stephen, *J. Chem. Soc.*, 1925, 1876.
Feulgen, Behrens, *Z. physiol. Chem.*, 1928, **177**, 229.
Gottfried, Ulzer, *Chem. Abstracts*, 1929, **23**, 1902.
Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1892.
Krafft, *Ber.*, 1880, **13**, 1416.

Palmitin.

See Tripalmitin.

α-Palmito-α′β-dilaurin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{11}H_{23}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{11}H_{23}$

$C_{43}H_{82}O_6$ MW, 694

Cryst. from EtOH. M.p. 47–8° (44·8°). n^{70} 1·43965. 100 ccs. EtOH dissolve 0·54 gm. at 23°. Sol. C_6H_6, $CHCl_3$, Et_2O.

Fischer, Bergmann, Bärwind, *Ber.*, 1920, **53**, 1605.
McElroy, King, *J. Am. Chem. Soc.*, 1934, **56**, 1192.

β-Palmito-αα′-dilaurin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{11}H_{23}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{11}H_{23}$

$C_{43}H_{82}O_6$ MW, 694

M.p. 47·8°. n^{70} 1·43980. 100 ccs. EtOH dissolve 0·38 gm. at 23°.

McElroy, King, *J. Am. Chem. Soc.*, 1934, **56**, 1192.

α-Palmito-α′β-dimyristin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{13}H_{27}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{13}H_{27}$

$C_{47}H_{90}O_6$ MW, 750

M.p. 53·0° (47·8°). Sol. Et_2O, $CHCl_3$, hot EtOH. 100 ccs. EtOH dissolve 0·42 gm. at 27·5°. 100 ccs. Me_2CO dissolve 1·82 gm. at 27·5°.

Averill, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.
Heiduschka, Schuster, *J. prakt. Chem.*, 1928, **120**, 153.

β-Palmito-αα′-dimyristin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{13}H_{27}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{13}H_{27}$

$C_{47}H_{90}O_6$ MW, 750

M.p. 59·8–60° (49·5°). Sol. Et_2O, $CHCl_3$, hot EtOH. 100 ccs. EtOH dissolve 0·10 gm. at 27·5. 100 ccs. Me_2CO dissolve 0·61 gm. at 27·5°.

Heiduschka, Schuster, *J. prakt. Chem.*, 1928, **120**, 152.
Averill, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.

α-Palmito-α′β-distearin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{17}H_{35}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{17}H_{35}$

$C_{55}H_{106}O_6$ MW, 862

Occurs in lard. Plates from C_6H_6–EtOH. M.p. 68·5° (63·2°, 62·6°). n_D^{70} 1·44245.

Amberger, Bromig, *Biochem. Z.*, 1922, **130**, 261.
Robinson, Roche, King, *J. Am. Chem. Soc.*, 1932, **54**, 708.
Bömer, *Chem. Zentr.*, 1913, I, 1620.

β-Palmito-αα′-distearin

$CH_2{\cdot}O{\cdot}CO{\cdot}C_{17}H_{35}$
$CH{\cdot}O{\cdot}CO{\cdot}C_{15}H_{31}$
$CH_2{\cdot}O{\cdot}CO{\cdot}C_{17}H_{35}$

$C_{55}H_{106}O_6$ MW, 862

Occurs in lard and in the fat of beef and mutton. Cryst. from Et_2O. M.p. 63·3° (67·9°, 64·8°, 68·0°). n_D^{70} 1·4467 (1·44374).

Kreis, Hafner, *Ber.*, 1903, **36**, 1124, 2766.
Bömer, Limprich, *Chem. Zentr.*, 1913, I, 1621.
Schuster, *J. pharm. chim.*, 1932, **16**, 421.
Amberger, Bromig, *Biochem. Z.*, 1922, **130**, 262.
Whitby, *J. Chem. Soc.*, 1926, 1461.
Robinson, Roche, King, *J. Am. Chem. Soc.*, 1932, **54**, 708.

Palmitoleic Acid (*Zoomaric acid*, 8-*hexadecenoic acid*, Δ^8-*hexadecylenic acid*, 8-*pentadecylene*-1-*carboxylic acid*)

$CH_3{\cdot}[CH_2]_5{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}COOH$

$C_{16}H_{30}O_2$ MW, 254

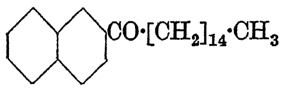
Found as glyceride in oil of whale, cod, walrus, seal, etc. α-Form, m.p. 32–3°. β-Form, m.p. 40–1°. B.p. 180–3°/1 mm. Dil. alk. $KMnO_4$ in cold ⟶ 8 : 9-dihydroxypalmitic acid.

Me ester: $C_{17}H_{32}O_2$. MW, 268. B.p. 140–1°/5 mm. H ⟶ methyl palmitate. $KMnO_4$ ⟶ heptylic and azelaic acids.

Et ester: $C_{18}H_{34}O_2$. MW, 282. Liq.

Armstrong, Hilditch, *J. Soc. Chem. Ind.*, 1925, **44**, 182T. (See also Hilditch, Vidyarthi, *J. Soc. Chem. Ind.*, 1927, **46**, 172T.)
Schmidt-Nielsen, *Chem. Zentr.*, 1922, I, 1047.
Toyama, *J. Soc. Chem. Ind. Japan*, 1927, **30**, 603.
Baudart, *Compt. rend.*, 1945, **220**, 404; *Chem. Abstracts*, 1946, **40**, 5398.

2-Palmitonaphthone (n-*Pentadecyl* β-*naphthyl ketone*)



$C_{26}H_{38}O$ MW, 366

Prisms from EtOH. M.p. 56°. B.p. 220°/0·03 mm.
Oxime: m.p. 77°.
Semicarbazone: m.p. 97°.

Buu-Hoï, Cagniant, *Bull. soc. chim.*, 1945, **12**, 307.

Palmitone (*Di-pentadecyl ketone, hentriacontanone*-16, 16-*ketohentriacontane*)

$CH_3\cdot[CH_2]_{13}\cdot CH_2\cdot CO\cdot CH_2\cdot[CH_2]_{13}\cdot CH_3$

$C_{31}H_{62}O$ MW, 450

Leaflets from EtOH. M.p. 82·8°. $D_4^{90\cdot9}$ 0·7947. Does not form bisulphite comp.
Oxime: needles from EtOH or AcOH. M.p. 59°.

Kipping, *J. Chem. Soc.*, 1890, **57**, 985.

Palmitophenone (*Pentadecyl phenyl ketone, palmitylbenzene*)

$C_6H_5\cdot CO\cdot[CH_2]_{14}\cdot CH_3$

$C_{22}H_{36}O$ MW, 316

Plates from EtOH. M.p. 59°. B.p. 250·5–251°/15 mm. Mod. sol. Et_2O. Prac. insol. cold EtOH. $D^{75\cdot9}$ 0·8692. $n_a^{75\cdot9}$ 1·46746. Triboluminescent. $CrO_3 \longrightarrow$ benzoic and pentadecylic acids.
Semicarbazone: m.p. 88°.

Krafft, *Ber.*, 1886, **19**, 2982.

Palmitylacetic Acid.
See 2-Ketostearic Acid.

***p*-Palmitylanisole.**
See under *p*-Hydroxypalmitophenone.

Palmitylglycine

$CH_3\cdot[CH_2]_{14}\cdot CO\cdot NH\cdot CH_2\cdot COOH$

$C_{18}H_{35}O_3N$ MW, 313

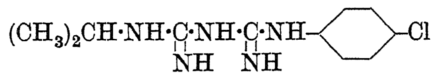
Cryst. from Me_2CO or EtOH. M.p. 125° (121°). Sinters at 119°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, hot C_6H_6. Insol. H_2O, pet. ether.
Me ester: $C_{19}H_{37}O_3N$. MW, 327. Cryst. from EtOH. M.p. 104–5°.
Et ester: $C_{20}H_{39}O_3N$. MW, 341. Needles from Et_2O. M.p. 80°.
Amide: plates from EtOH. M.p. 161–2°.

Bondi, Frankl, *Biochem. Z.*, 1909, **17**, 552.
Abderhalden, Funk, *Z. physiol. Chem.*, 1910, **65**, 62.
Karrer, Miyamichi, Storm, Widmer, *Helv. Chim. Acta.*, 1925, **8**, 205.
Koebner, *J. Chem. Soc.*, 1941, 564.

***p*-Palmitylphenetole.**
See under *p*-Hydroxypalmitophenone.

***p*-Palmitylphenol.**
See *p*-Hydroxypalmitophenone.

Paludrine (N[1]-p-*Chlorophenyl*-N[5]-*isopropyldiguanide, Proguanil B.P.C., Chlorguanide*)



$C_{11}H_{16}N_5Cl$ MW, 253·5

Colourless cryst. from EtOH.Aq. M.p. 130–1°.
Hydrochloride: colourless cryst. M.p. 244–5°. Spar. sol. cold H_2O.
Acetate: colourless cryst. from Me_2CO. M.p. 188–9°.
Antimalarial drug administered in the form of a salt. A true causal prophylactic in malignant tertian malaria, in most of strains of which it achieves a permanent cure; and partial causal prophylactic in benign tertian malaria.

Curd, Rose, *J. Chem. Soc.*, 1946, 729.
Curd, Davy, Rose, *Ann. Trop. Med. and Parasit.*, 1945, **39**, 208.
I.C.I., B.P.s 577,843; 603,069; 607,720.

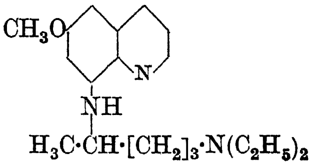
Palustrin

$C_{17}H_{29}O_2N_3$ MW, 307

Alkaloid isolated from swamp horsetail (*Equisetum palustre*). Cryst. M.p. 121°. $[\alpha]_D^{18}$ +15·8° in $CHCl_3$. Contains 2 active H atoms and 1 ethylenic linkage, but no methoxyl group. Tetranitromethane $\longrightarrow$ yellow col.
B,HCl: cryst. M.p. 180–90° decomp. $[\alpha]_D^{18}$ + 8·3° in H_2O.
Picrate: yellow prisms. M.p. 150°.
Chloroplatinate: bright orange cryst. M.p. 221–3° decomp.

Karrer, Eugster, *Helv. Chim. Acta*, 1948, **31**, 1062.

Pamaquin (*Plasmoquin*, N-(4-*diethylamino*-1-*methylbutyl*)-6-*methoxy*-8-*aminoquinoline*)



$C_{19}H_{29}ON_3$ MW, 315

B.p. 185–7°/0·2 mm. Antimalarial drug. Used, in the form of a salt, in the radical cure of benign tertian malaria. Pamaquin B.P. is the salt with 2 : 2′-dihydroxy-1 : 1′-dinaphthylmethane-3 : 3′-dicarboxylic acid.
Citrate : m.p. 125–7° decomp.
Di-meconate : m.p. 175–8° decomp.

Elderfield *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 1516.
Bayer, B.P. 267,169.
I.G., D.R.P. 486,079.

Panax sapogenin.
See Oleanolic Acid.

***dl*-Pantamide** (1 : 3-*Dihydroxy-2-dimethylbutyramide*)

$HO{\cdot}CH_2{\cdot}C(CH_3)_2{\cdot}CH(OH){\cdot}CONH_2$

$C_6H_{13}O_3N$ MW, 147

Prisms from Me_2CO. M.p. 127–8°.

Madinaveitia, Martin, Rose, Swain, *Biochem. J.*, 1945, **39**, 85.

Pantetheine (Lactobacillus bulgaricus *Factor*)

CH_3
$HO{\cdot}CH_2{\cdot}C{\cdot}CH(OH){\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}NH$
CH_3 CH_2
CH_2SH

$C_{11}H_{22}O_4N_2S$ MW, 278

Growth factor for *Lactobacillus bulgaricus*. Resin. $[\alpha]^{20}_D$ + 12·9° in H_2O.

McRorie, Masley, Williams, *Arch. Biochem.*, 1950, **27**, 471.
Brown, Craig, Snell, *ibid.*, 473.
Snell *et al.*, *J. Am. Chem. Soc.*, 1950, **72**, 5349.
Baddiley, Thain, *Chemistry and Industry*, 1951, 337; *J. Chem. Soc.*, 1951, 2253; 1952, 800.

Pantocaine.
See Decicaine.

Pantoic Acid (1 : 3-*Dihydroxy-2-dimethylbutyric acid*)

$HO{\cdot}CH_2{\cdot}C(CH_3)_2{\cdot}CH(OH){\cdot}COOH$

$C_6H_{12}O_4$ MW, 148

d-.
Brucine salt : m.p. 212–14°. $[\alpha]^{25}_D$ −3·25°.
Quinine salt : m.p. 164–5°.

l-.
Quinine salt : m.p. 183°.

dl-.
Amide : *see* Pantamide.

Kuhn, Wieland, *Ber.*, 1940, **73**, 962, 971.
Beutel, Tishler, *J. Am. Chem. Soc.*, 1946, **68**, 1463.

Pantothenic Acid

CH_3
$HO{\cdot}CH_2{\cdot}C{\cdot}CH(OH){\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$
CH_3

$C_9H_{17}O_5N$ MW, 219

d-.
Growth-promoting and antidermatitic factor ("Filtrate factor") of vitamin B complex, present in living cells particularly of the liver. Viscous oil. $[\alpha]^{25}_D$ + 37·5° in H_2O. Acid or alk. hyd. ⟶ β-alanine + *l*-α-hydroxy-ββ-dimethyl-γ-butyrolactone (or salt of corresponding acid).
Na salt : cryst. from EtOH. M.p. 122–4° (121–2°). $[\alpha]^{12·5}_D$ + 29·5° in H_2O, $[\alpha]^{25}_D$ + 27·04° in H_2O.
Ca salt : cryst. from MeOH. M.p. 195–6°. $[\alpha]^{26}_D$ + 28·2° in H_2O. Anisotropic.
Benzylthiouronium salt : m.p. 148–9°.
Cinchonidine salt : m.p. 178–9° (176–7°). $[\alpha]^{28·4}_D$ − 60·6° in MeOH, $[\alpha]^{18}_D$ − 62·8° in H_2O.
Quinine salt : m.p. 136–7°. $[\alpha]^{19}_D$ − 95° in H_2O.
Me ester : $C_{10}H_{19}O_5N$. MW, 233. Oil. $[\alpha]^{15}_D$ + 37·1° in Me_2CO.
Et ester : $C_{11}H_{21}O_5N$. MW, 247. Oil. $[\alpha]^{18}_D$ + 36·8° in EtOH.

l-.
Little or no biological activity (conflicting results). Viscous oil. $[\alpha]^{21}_D$ − 26·7° in H_2O, − 56·3° in MeOH.
Na salt : m.p. 120–2°. $[\alpha]^{15}_D$ − 27·4° in H_2O.
Ca salt : cryst. from MeOH. M.p. 187·5–189°. $[\alpha]^{26}_D$ − 27·8° in H_2O.
Benzylthiouronium salt : m.p. 150–1° (145–6°).
Quinine salt : needles from Me_2CO–MeOH. M.p. 183·0° (167–8°). $[\alpha]^{19}_D$ − 121° in H_2O.
Et ester : oil. $[\alpha]^{19}_D$ − 37·3° in EtOH.

dl-.
Viscous oil. Possesses half biological activity of *d-* form.
Et ester : b.p. 118–20°/10^{-3} mm. n^{17}_D 1·477.
Benzylthiouronium salt : needles from Me_2CO. M.p. 135–6°.

Woolley *et al.*, *J. Biol. Chem.*, 1939, **129**, 673.
Williams *et al.*, *J. Am. Chem. Soc.*, 1940, **62**, 1776, 1784.
Stiller *et al.*, *J. Am. Chem. Soc.*, 1940, **62**, 1779, 1785; 1941, **63**, 1237, 2846.
Woolley, *J. Am. Chem. Soc.*, 1940, **62**, 2251.
Reichstein *et al.*, *Helv. Chim. Acta*, 1940, **23**, 650, 1276; 1941, **24**, 185.
Kuhn *et al.*, *Ber.*, 1940, **73**, 971, 1134; 1941, **74**, 218.
Parke, Lawson, *J. Am. Chem. Soc.*, 1941, **63**, 2869.
Harris, Boyack, Folkers, *J. Am. Chem. Soc.*, 1941, **63**, 2662.
Michi, Funabashi, *Chem. Abstracts*, 1947, **41**, 6200.

Pantoyltaurine (N-(1 : 3-*Dihydroxy-2-dimethylbutyryl*)-*taurine*)

$HO\cdot CH_2\cdot C(CH_3)_2\cdot CH(OH)\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot SO_3H$

$C_8H_{17}O_6NS$ MW, 255

d-.

Na salt: $[\alpha]_D^{22}$ + 23·2° in H_2O.

Quinine salt: m.p. 160–2°. $[\alpha]_D^{22}$ − 76·2° in H_2O.

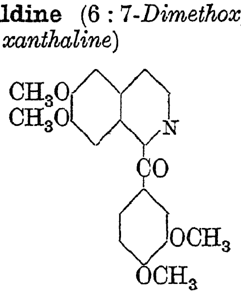
l-.

Na salt: $[\alpha]_D^{22}$ − 25·3° in H_2O.

Quinine salt: m.p. 179–179·5°. $[\alpha]_D^{22}$ − 99·4° in H_2O.

Kuhn, Wieland, Möller, *Ber.*, 1941, **74**, 1605.

Papaveraldine (6 : 7-*Dimethoxy*-1-*veratroylisoquinoline, xanthaline*)



$C_{20}H_{19}O_5N$ MW, 353

Occurs in opium. Cryst. from C_6H_6 or pet. ether. M.p. 210° (208°). Sol. min. acids, hot AcOH. Mod. sol. C_6H_6, $CHCl_3$. Spar. sol. EtOH, Et_2O, pet. ether. Insol. H_2O. Zn + H_2SO_4 or AcOH ⟶ papaverinol. Zn + Ac_2O ⟶ papaverine. HNO_3 ⟶ nitropapaveraldine. KOH fusion ⟶ veratric acid + 6 : 7-dimethoxyisoquinoline.

B,HCl,2½(?)H₂O: yellow cryst. from H_2O.

B,H₂SO₄: yellow needles. Decomp. by H_2O.

B,HNO₃,2H₂O: yellow needles. Decomp. by hot H_2O.

B₂,H₂PtCl₆,H₂O: orange-red prisms from dil. HCl. Decomp. at 210°.

Picrate: yellow needles. M.p. 208–9°.

Methiodide, H₂O: yellow prisms. M.p. 194°.

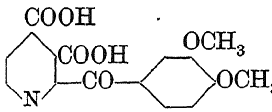
Methiodide, 3H₂O: yellow prisms. M.p. 136° (132°).

Oxime: exists in two forms. (i) Needles or prisms. M.p. 235–6°; (ii) prisms from EtOH. M.p. 245°.

Phenylhydrazone: reddish-yellow aggregates from dil. EtOH. M.p. 80–1°.

Stuchlik, *Monatsh.*, 1900, **21**, 828.
Pschorr, *Ber.*, 1904, **37**, 1936.
Dobson, Perkin, *J. Chem. Soc.*, 1911, **99**, 135.
Buck, Haworth, Perkin, *J. Chem. Soc.*, 1924, **125**, 2184.
Menon, *Proc. Indian Acad. Sci.*, 1944, **19***A*, 21.

Papaveric Acid (2-*Veratroylcinchomeronic acid*)



$C_{16}H_{13}O_7N$ MW, 331

Cryst. + H_2O from H_2O. M.p. 233° decomp. Sol. hot dil. EtOH, AcOH, dil. HCl. Spar. sol. cold H_2O, EtOH, Et_2O, Me_2CO, $CHCl_3$, CS_2, C_6H_6, pet. ether. Sol. conc. H_2SO_4 with reddish-yellow col. KOH fusion ⟶ protocatechuic acid. Ac_2O ⟶ papaveric anhydride. HNO_3 ⟶ nitropapaveric acid.

K₂A,2½H₂O: plates from dil. EtOH. Sol. H_2O. Spar. sol. EtOH.

B,HCl: orange-red cryst. from HCl.Aq.

B,HCl,2½H₂O: yellow needles.

Oxime: needles from EtOH. M.p. 154–7°. Sol. H_2O, EtOH.

Phenylhydrazone: yellow needles from EtOH. M.p. 190°.

3-*Me ester*: $C_{17}H_{15}O_7N$. MW, 345. Cryst. from MeOH. M.p. 156°. Sol. MeOH. Spar. sol. H_2O.

4-*Me ester*: needles from H_2O or MeOH. M.p. 198° decomp. (196°). Sol. EtOH, Me_2CO, AcOEt. Spar. sol. H_2O, MeOH. Insol. Et_2O.

Di-Me ester: $C_{18}H_{17}O_7N$. MW, 359. Plates from Me_2CO. M.p. 121–2° (122–4°). Sol. Me_2CO, AcOEt.

3-*Et ester*: $C_{18}H_{17}O_7N$. MW, 359. Needles. M.p. 187–8°. Spar. sol. H_2O.

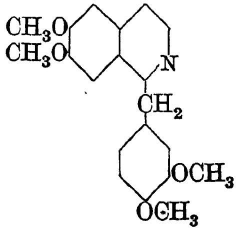
4-*Et ester*: needles from EtOH. M.p. 184°. Spar. sol. H_2O.

Anhydride: $C_{32}H_{24}O_{13}N_2$. MW, 644. Needles from C_6H_6. M.p. 169–70°.

Methochloride, H₂O: yellow plates. M.p. 182–4° decomp.

Goldschmiedt, *Monatsh.*, 1885, **6**, 380; 1888, **9**, 357.
Goldschmiedt, Strache, *Monatsh.*, 1889, **10**, 159, 692.
Goldschmiedt, Kirpal, *Monatsh.*, 1896, **17**, 496.
Kirpal, *Monatsh.*, 1897, **18**, 464.
Schranzhofer, *Monatsh.*, 1893, **14**, 525.
Wegscheider, *Monatsh.*, 1902, **23**, 338, 388.
Goldschmiedt, Honigschmid, *Monatsh.*, 1903, **24**, 681.

Papaverine (6 : 7-*Dimethoxy*-1-*veratryliso-quinoline, papaveroline tetramethyl ether*)



$C_{20}H_{21}O_4N$ **MW, 339**

Occurs in opium (0·8–1·0%). Prisms from EtOH–Et_2O. Needles from $CHCl_3$–pet. ether. M.p. 147°. Sol. hot EtOH, $CHCl_3$, Me_2CO. Mod. sol. hot C_6H_6. Prac. insol. H_2O, pet. ether. 1 part dissolves in 86 parts 97% EtOH at 15° and in 258 parts Et_2O at 10°. Sol. conc. H_2SO_4 colourless, changing to violet on warming. D_4 1·308–1·337. Optically inactive. Triboluminescent. $Na_2Cr_2O_7$ ⟶ papaveraldine. $KMnO_4$ ⟶ papaveraldine, papaveric acid, 6 : 7-dimethoxyisoquinoline-1-carboxylic acid, pyridine-2 : 3 : 4-tricarboxylic acid, metahemipinic acid, veratric acid, oxalic acid, CO_2 and NH_3. Sn + HCl ⟶ tetrahydropapaverine. Br ⟶ bromopapaverine. HNO_3 ⟶ nitropapaverine. Nitrous fumes ⟶ nitrosopapaverine. HNO_2 ⟶ papaveraldoxime, m.p. 235–6°. Boiling conc. HCl ⟶ papaveroline dimethyl ether. HI ⟶ papaveroline. Dist. with KOH ⟶ methylamine, protocatechuic acid, homocatechol dimethyl ether and oxalic acid.

B,HCl: plates from H_2O. M.p. 220–1° decomp. (210–13° decomp.). Sol. 37·3 parts H_2O at 18°.

B,HBr: cryst. from H_2O or dil. EtOH. M.p. 213–14° decomp.

B,HI: m.p. 200° decomp. Sol. hot H_2O, EtOH. Dimorphous.

B,HFeCl$_4$,H$_2$O: red plates. M.p. 195°.

B,HFeCl$_4$,2H$_2$O: orange prisms. M.p. 195°.

B$_2$,H$_2$PtCl$_6$,½H$_2$O: m.p. 186°.

B$_2$,H$_2$PtCl$_6$,2H$_2$O: orange prisms from conc. HCl. M.p. 198°. Insol. H_2O, EtOH, Et_2O.

Acid oxalate: needles or prisms from H_2O. M.p. 196°. Sol. hot H_2O. Spar. sol. hot EtOH.

Succinate: plates from EtOH. M.p. 171°. Sol. hot H_2O.

Salicylate: plates from EtOH. M.p. 130°.

Benzoate: cryst. from EtOH. M.p. 145°. Sol. EtOH. Insol. H_2O.

Methiodide, 4H$_2$O: cryst. from dil. EtOH. M.p. 60–5° (55–60°), 195° anhyd. Sol. $CHCl_3$. Mod. sol. C_6H_6. Insol. Et_2O.

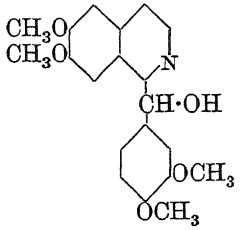
Ethiodide: yellow cryst. from dil. EtOH. M.p. 216° decomp. Sol. $CHCl_3$. Spar. sol. C_6H_6. Insol. Et_2O.

Picrate: yellow plates from EtOH. M.p. 183° (179° decomp., 154°).

Picrolonate: needles from EtOH. M.p. 221°.

Pictet, Gams, *Ber.*, 1909, **42**, 2943.
Buck; Haworth, Perkin, *J. Chem. Soc.*, 1924, **125**, 2179.
Rosenmund, Nothnagel, Riesenfeldt, *Ber.*, 1927, **60**, 392.
Späth, Burger, *ibid.*, 704.
Mannich, Walther, *Arch. Pharm.*, 1927, **265**, 1.
Kindler, Peschke, *Arch. Pharm.*, 1934, **272**, 236.

Papaverinol



$C_{20}H_{21}O_5N$ MW, 355

Prisms from dil. MeOH. M.p. 137°. Sol. MeOH, EtOH, Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. H_2O, Et_2O. Ox. ⟶ papaveraldine.

B,HCl: yellow needles from EtOH–Et_2O. M.p. 200–2°.

B$_2$,H$_2$PtCl$_6$: orange-yellow needles from dil. HCl. M.p. 168° decomp.

O-*Benzoyl*: amorph. Sol. EtOH, $CHCl_3$, AcOH. *Picrate*: yellow needles from EtOH. M.p. 126° decomp.

O-p-*Bromobenzoyl*: cryst. M.p. 194°.

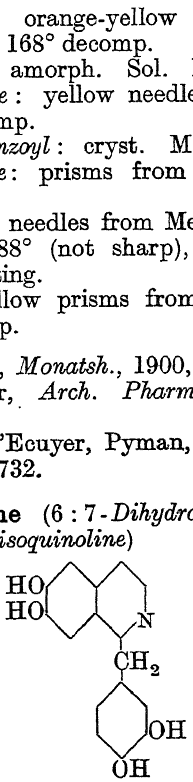
Methochloride: prisms from EtOH. M.p. 205–6°.

Methiodide: needles from MeOH or EtOH–Et_2O. M.p. 188° (not sharp), 200° decomp. with rapid heating.

Picrate: yellow prisms from EtOH. M.p. 168–71° decomp.

Stuchlik, *Monatsh.*, 1900, **21**, 814.
Gadamer, *Arch. Pharm.*, 1915, **253**, 284.
King, L'Ecuyer, Pyman, *J. Chem. Soc.*, 1936, 732.

Papaveroline (6 : 7-*Dihydroxy*-1-[3 : 4-*dihydroxybenzyl*]-*isoquinoline*)



$C_{16}H_{13}O_4N$ MW, 283

Cryst. powder + $2H_2O$. Loses H_2O at 100°. Darkens at 150° and decomp. on further heating without melting. Sol. AcOH, glycerol. Mod. sol. EtOH. Spar. sol. Et_2O, $CHCl_3$. Insol. H_2O, C_6H_6, pet. ether. Sol. dil. min. acids. Dist. with Zn dust ⟶ 1-methylisoquinoline.

B,HCl,H$_2$O: needles. Sol. hot H_2O.

B,HI,2H$_2$O: needles from H_2O. M.p. anhyd. 230° decomp. Mod. sol. hot H_2O.

B$_2$,H$_2$SO$_4$,10H$_2$O: loses H_2O at 105°.

B$_2$,(COOH)$_2$,3H$_2$O: needles. Sol. hot H_2O.

Methochloride: m.p. 264° decomp. (235°).

Ethochloride: cryst. M.p. 215°.

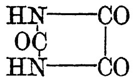
Benzylchloride: m.p. 158°.
Methiodide: m.p. 208° decomp. From dil. HCl m.p. 77° (probably hydrated).
Methopicrate: m.p. 210° decomp.
Tetra-Me ether: *see* Papaverine.
O-*Tetrabenzoyl*: m.p. 148°.

Goldschmiedt, *Monatsh.*, 1885, **6**, 967.
Krauss, *Monatsh.*, 1890, **11**, 350.
Claus, Kassner, *J. prakt. Chem.*, 1897, **56**, 344.
Kitasato, Robinson, *J. Chem. Soc.*, 1932, 785.

Para-acetaldehyde.
See Paraldehyde.

Para-anthracene.
See Dianthracene.

Parabanic Acid (*Oxalylurea*)



$C_3H_2O_3N_2$ MW, 114

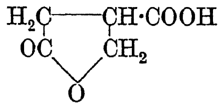
Needles or prisms from boiling H_2O. Part. sublimes at 100°. Decomp. at 243°. Sol. 21 parts H_2O at 8°. Decomp. by boiling aq. alkalis. Salts unstable.
$C_3H_2O_3N_2,H_2O$: cryst. Sol. 7·4 parts H_2O at 8°. Loses H_2O at 150–60°.

Behrend, Asche, *Ann.*, 1918, **416**, 226.
Biltz, Schiemann, *Ber.*, 1926, **59**, 721.

Paracasein.
See Caseinogen.

Parachloralose.
See β-Glucochloralose.

Paraconic Acid (*5-Ketotetrahydrofuran-3-carboxylic acid, butyrolactone-β-carboxylic acid, 5-ketotetrahydro-β-furoic acid*)



$C_5H_6O_4$ MW, 150

Cryst. M.p. 57–8° (55°). Dist. ⟶ citraconic anhydride.

Fittig, Beer, *Ann.*, 1883, **216**, 84.
Reitter, *Ber.*, 1898, **31**, 2724.

Paracoumarone.
See Coumarone.

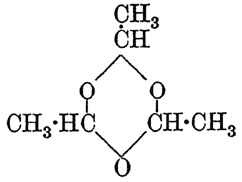
Paracyanoformic Acid.
See Triazine-tricarboxylic Acid.

Paraformaldehyde.
See under Formaldehyde.

Para-indene.
See Indene.

Paralactic Acid.
See Lactic Acid.

Paraldehyde (*Para-acetaldehyde, 2 : 4 : 6-trimethyl-1 : 3 : 5-trioxan*)



$C_6H_{12}O_3$ MW, 132

M.p. 12·6° (10°). B.p. 128° (124°). Spar. sol. H_2O. D_4^{10} 1·0037, D_4^{15} 0·9975, D_4^{20} 0·9943, $D_4^{35·5}$ 0·9772, $D_4^{53·7}$ 0·9536, D_4^{75} 0·9264, $D_4^{124·3}$ 0·8738. n_D^{20} 1·4049. Heat of comb. C_p = 813·2 Cal. Gives acetaldehyde on warming with a few drops of conc. H_2SO_4, or on boiling with dil. HCl. Ac_2O + conc. H_2SO_4 ⟶ ethylidene diacetate. PCl_5 ⟶ ethylidene chloride. Br water ⟶ acetic acid. Br in $CHCl_3$ ⟶ bromo- and dibromo-acetaldehyde.

Friedel, *Bull. soc. chim.*, 1893, **9**, 385.
Geuther, Cartmell, *Ann.*, 1859, **112**, 16.
Vogt, Nieuwland, *J. Am. Chem. Soc.*, 1921, **43**, 207B.
Morton, Nicolet, U.S.P., 1,300,451, (*Chem. Abstracts*, 1919, **13**, 1861).
Hunt, Reuter, *Chem. Abstracts*, 1949, **43**, 7193.

Paraldol (*Dimeride of aldol*)

$C_8H_{16}O_4$ MW, 176

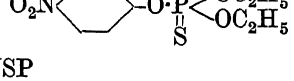
Prisms. M.p. 90° (82°, 80–90°). B.p. 90–100° in high vacuo. D^{20} 1·116. n_D^{20} 1·4610. Sol. H_2O, EtOH. Less sol. Et_2O. Vac. dist. ⟶ aldol. Ox. ⟶ 2-hydroxybutyric acid. Heat in presence of I ⟶ crotonaldehyde. H(+ Ni) ⟶ β-butylene glycol.

Nowak, *Monatsh.*, 1901, **22**, 1142.
Wurtz, *Compt. rend.*, 1883, **97**, 1525.
Späth, Schmid, *Ber.*, 1941, **74**, 859.
Späth, Lorenz, Freund, *Ber.*, 1942, **75**, 1029; *Ber.*, 1943, **76**, 57.
Späth, Meinhard, *Ber.*, 1943, **76**, 504.
Conolly, *J. Chem. Soc.*, 1943, 42.

Paranthrene.
See Dianthracene.

Parasiticin.
See Penicillin-G.

Parathion (*Diethyl* p-*nitrophenyl thiophosphate, Thiophos* 3422)



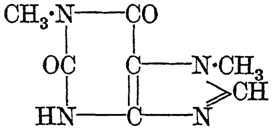
$C_{10}H_{14}O_5NSP$ MW, 291

Pale yellow oil. B.p. 157–62°/0·6 mm. n_D^{25} 1·5370. Insecticide.

Fletcher *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 3943.

Paratophan.
See 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

Paraxanthine (1 : 7-*Dimethylxanthine*, 1 : 7-*dimethyl*-2 : 6-*dihydroxypurine*)



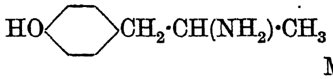
$C_7H_8O_2N_4$ MW, 180

Present in human urine. Plates from H_2O. M.p. 298–9° (289°, 284°). Sol. NH_3.Aq., HCl + HNO_3. Spar. sol. cold H_2O. Mod. sol. hot H_2O. Insol. EtOH, Et_2O, CH_3I ⟶ caffeine.

B,HCl,H₂O : dissociates at 100°. Decomp. in H_2O.
B_2,H_2PtCl_6,H_2O : orange cryst.
$B,HAuCl_4,\frac{1}{2}H_2O$: m.p. 227–8°.
Picrate : yellow ppt. Decomp. in H_2O.

Fischer, *Ber.*, 1897, **30**, 554, 2400.
Fischer, Clemm, *Ber.*, 1898, **31**, 2623.
Fischer, Ach, *Ber.*, 1906, **39**, 423.
Traube, Dudley, *Ber.*, 1913, **46**, 3839.
Boehringer, Sohn, D.R.P., 582,435, (*Chem. Abstracts*, 1933, **27**, 5754); D.R.P., 576,604, (*Chem. Abstracts*, 1933, **27**, 5757).

Paredrine (1-p-*Hydroxyphenylisopropylamine*, p-β-*aminopropylphenol*)



$C_9H_{13}ON$ MW, 151

Exerts marked pressor activity similar to that of benzedrine.

B,HCl : cryst. from EtOH–Et_2O. M.p. 171–2°. Sol. H_2O, Et_2O.

Alles, *J. Am. Chem. Soc.*, 1932, **54**, 271.
Woodruff, Conger, *J. Am. Chem. Soc.*, 1938, **60**, 465.
Iglauer, Altschule, *American Journal of Medical Sciences*, 1940, **199**, 359, (*Chem. Abstracts*, 1940, **34**, 3821).
Hoover, Hass, *J. Org. Chem.*, 1947, **12**, 501.

Parietin.
See Physcione.

Parigenin.
See Sarsasapogenin.

Parinaric Acid

$CH_3{\cdot}CH_2{\cdot}[CH{:}CH]_4{\cdot}[CH_2]_7{\cdot}COOH$

$C_{18}H_{28}O_2$ MW, 276

Acid of the kernel fat of *Parinarium laurinum*. Faintly yellow plates from pet. ether. M.p. 85–6°. Isomerised by I or light to acid, m.p. 95–6°. Absorption maxima: 3200, 3070 and 2920 Å. in EtOH.

Farmer, Sunderland, *J. Chem. Soc.*, 1935, 759.
Kaufmann, Baltes, Funke, *Chem. Zentr.*, 1938, II, 4143.

Patchoulene.
See Patschulene.

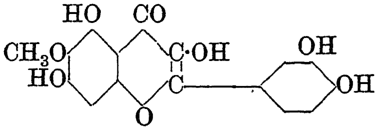
Patschulene (*Patchoulene*)

$C_{15}H_{24}$ MW, 204

Liq. with cedarwood odour. B.p. 255–6°, 252–3°/743 mm., 112–15°/12–12·5 mm. D_4^{20} 0·9296. n_D^{20} 1·49853. $[\alpha]_D^{20}$ − 38·08°. Very sol. Et_2O, C_6H_6. Spar. sol. EtOH, AcOH. Insol. HCl, HNO_3, H_2SO_4, but gives red col. with these acids.

Wallach, Tuttle, *Ann.*, 1894, **279**, 394.
Schimmel, *Chem. Zentr.*, 1904, I, 1265.

Patuletin (6-O-*Methylquercetagetin*)



$C_{16}H_{12}O_8$ MW, 332

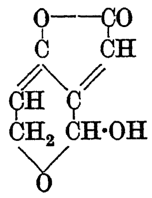
Colouring matter of flowers of *Tagetes patula*. Pale yellow needles from EtOH.Aq. M.p. 262–4°. Alk. ox. ⟶ protocatechuic acid.

Penta-acetyl : cryst. from AcOH.Aq. M.p. 170–2°.

Penta-Me ether : hexa-Me ether of Quercetagetin. *See under* Quercetagetin.

Rao, Seshadri, *Proc. Indian Acad. Sci.*, 1941, **14A**, 643, (*Brit. Chem. Abstracts*, 1942, A II, 202).
Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23A**, 140, (*Chem. Abstracts*, 1947, **41**, 121).

Patulin (*Clavacin, clavatin*)

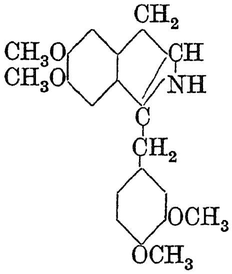


$C_7H_6O_4$ MW, 154

Cryst. from $CHCl_3$. M.p. 109·5–110·5°.
Acetate : m.p. 118–20°.
Phenylhydrazone : m.p. 149–50°.

Birkinshaw, Bracken, Michael, Raistrick, *Lancet*, 1943, **245**, 625.
Bergel, Morrison, Moss, Rinderknecht, *J. Chem. Soc.*, 1944, 415.
Woodward, Singh, *J. Am. Chem. Soc.*, 1949, **71**, 758; 1950, **72**, 1428.

Pavine (2 : 4-*Dihydropapaverine*)



$C_{20}H_{23}O_4N$ MW, 341

d-.

Cryst. from C_6H_6. M.p. 224°. $[\alpha]_D^{23} + 150{\cdot}3°$ in $CHCl_3$, $+ 198{\cdot}8°$ in AcOH.

d-*Tartrate*: cryst. from Me_2CO–Et_2O. M.p. 156–8° decomp. Very sol. H_2O, org. solvents. $[\alpha]_D^{22} + 157{\cdot}5°$.

l-.

Cryst. from C_6H_6. M.p. 224°. $[\alpha]_D^{23} - 150{\cdot}8°$ in $CHCl_3$.

d-*Tartrate*: cryst. from Me_2CO–Et_2O. M.p. 156–8° decomp. Very sol. H_2O, org. solvents. $[\alpha]_D^{22} - 150{\cdot}5°$ in H_2O.

dl-.

Prisms from xylene, needles from EtOH. M.p. 201–2°. Sol. $CHCl_3$, CS_2, warm Me_2CO, C_6H_6. Spar. sol. Et_2O, pet. ether.

B,HCl: cryst. $+ 5H_2O$. Decomp. at 325°.

N-*Me*: $C_{21}H_{25}O_4N$. MW, 355. Prisms from Et_2O or C_6H_6. M.p. 140–1°. Sol. most org. solvents. Spar. sol. H_2O, Et_2O, pet. ether. Conc. $H_2SO_4 \longrightarrow$ weak yellowish-green col. *B,HCl*: prisms $+ 8H_2O$ from H_2O. M.p. 65–81°. *B,HI*: prisms $+ 6H_2O$ from H_2O. M.p. 70–87°. *Picrate*: yellow needles from H_2O. M.p. 219°. Spar. sol. H_2O, EtOH. *Methiodide*: prisms $+ 2H_2O$ from MeOH. M.p. about 280°. Spar. sol. H_2O.

N-*Benzoyl*: prisms from EtOH. M.p. 234–5°. Sol. $CHCl_3$. Spar. sol. EtOH, Me_2CO. Very spar. sol. H_2O, Et_2O.

N-*Nitroso*: prisms from EtOH. M.p. 180–2°. Sol. warm EtOH. Mod. sol. $CHCl_3$, Me_2CO, C_6H_6. Spar. sol. Et_2O. Insol. pet. ether.

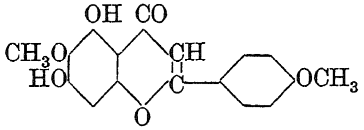
Picrate: yellow needles. Decomp. at 285°.

Pyman, *J. Chem. Soc.*, 1909, **95**, 1620; 1915, **107**, 177.
Pope, Gibson, *J. Chem. Soc.*, 1910, **97**, 2209.
Pyman, Reynolds, *ibid.*, 1324.

Pectenin.
See Octopin.

Pectenine.
See Carnegine.

Pectolinarigenin (5 : 7-*Dihydroxy*-6 : 4′-*dimethoxyflavone*)



$C_{17}H_{14}O_6$ MW, 314

Yellow needles from EtOH or dil. Me_2CO. M.p. 215–16°.

Diacetyl: needles. M.p. 151°.

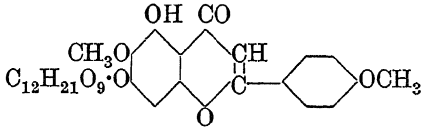
7-*Me ether*: $C_{18}H_{16}O_6$. MW, 328. Yellow needles. M.p. 188°. *Acetyl*: m.p. 188°.

Di-Me ether: $C_{19}H_{18}O_6$. MW, 342. M.p. 162°.

Glycoside: *see* Pectolinarin.

Merz, Wu, *Arch. Pharm.*, 1936, **274**, 126.
Robinson, Schwarzenbach, *J. Chem. Soc.*, 1930, 829.

Pectolinarin



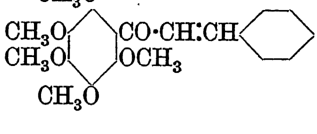
$C_{29}H_{34}O_{15}$ MW, 622

A glycoside from the flowers of *Linaria vulgaris*, Linn. M.p. 240–50° decomp. Hyd. by conc. HCl to glucose, rhamnose and pectolinarigenin.

Hepta-acetyl: cryst. from Et_2O–pet. ether. M.p. 134–8°. $[\alpha]_D^{18} - 68{\cdot}5°$.

Merz, Wu, *Arch. Pharm.*, 1936, **274**, 126.
Zemplén, Bognár, *Ber.*, 1941, **74**, 1818.
Zemplén, Bognár, Mester, *Ber.*, 1942, **75**, 489.
Zemplén, Farkas, *Ber.*, 1943, **76**, 937.

Pedicellin



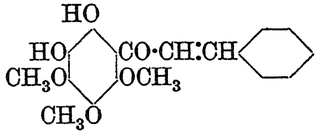
$C_{20}H_{22}O_6$ MW, 358

Isolated from dried leaves of *Didymocarpus pedicellata*. Cryst. from Et_2O. M.p. 93°. $HNO_3 \longrightarrow$ methylpedicinin ($C_{17}H_{14}O_6$), m.p. 110°.

Phenylhydrazone: rods from AcOEt. M.p. 133–5°.

Sharma, Siddiqui, *J. Indian Chem. Soc.*, 1939, **16**, 1.
Bose, Dutt, *J. Indian Chem. Soc.*, 1940, **17**, 499.
Baker, *J. Chem. Soc.*, 1941, 662.

Pedicin



Suggested structure

$C_{18}H_{18}O_6$ MW, 330

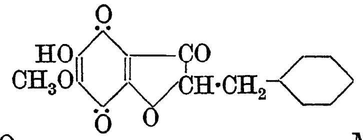
Isolated from coarsely powdered leaves of *Didymocarpus pedicellata*. Orange red plates. M.p. 145°. Br in $CHCl_3$ or $KMnO_4$ ⟶ pedicinin. Methylation ⟶ pedicellin.

Dibenzoyl: rods from C_6H_6. M.p. 181–3°.

Phenylhydrazone: needles from EtOH. M.p. 165–7°.

Siddiqui *et al.*, *J. Indian Chem. Soc.*, 1937, **14**, 703; 1939, **16**, 1, 423.

Pedicinin



$C_{16}H_{12}O_6$ MW, 300

Isolated from dried leaves of *Didymocarpus pedicellata*. Red rods or needles from C_6H_6. M.p. 203°. Alkalis ⟶ benzaldehyde.

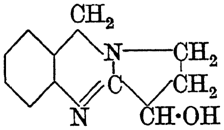
Monoacetyl: prisms from AcOEt. M.p. 175°.

Siddiqui, *J. Indian Chem. Soc.*, 1937, **14**, 703.

Bose, Dutt, *J. Indian Chem. Soc.*, 1940, **17**, 499.

Salooja, Sharma, Siddiqui, *Chem. Abstracts*, 1948, **42**, 1930.

Peganine (*Vasicine*)



$C_{11}H_{12}ON_2$ MW, 188

l-.

Found in *Adhatoda vasica*, N. Cryst. from EtOH. M.p. 211–12°. $[\alpha]_D^{14}$ − 254° in $CHCl_3$, − 61·5° in EtOH.

dl-.

Needles from EtOH. M.p. 209–10° (196°). Sol. EtOH, $CHCl_3$. Spar. sol. H_2O, Et_2O, C_6H_6.

B,HCl: needles + $2H_2O$. M.p. anhyd. 208°.

B,HI: needles + $2H_2O$. M.p. anhyd. 195°.

Methiodide: needles from MeOH. M.p. 187°.

Acetyl deriv.: m.p. 122–3°. B.p. 230–40°/0·01 mm.

Picrate: needles. M.p. 199° decomp.

Späth, Kuffner, Platzer, *Ber.*, 1935, **68**, 497, 699, 1384.

Späth, Platzer, *Ber.*, 1936, **69**, 255, 384.

Narang, Ray, *J. Chem. Soc.*, 1936, 686.

Späth, *Monatsh.*, 1938, **72**, 115.

Peimine

$C_{26}H_{43}O_3N$ MW, 417

Alkaloid from *Fritillaria Roylei*, Stuart (Pei-Mu). M.p. 224°.

B,HCl: m.p. 295° decomp.

B,HBr: m.p. 293·5–294°.

B,H₂SO₄: m.p. 278–80°.

B₂,H₂PtCl₆: m.p. 233–5° decomp.

B,HAuCl₄: m.p. 164–5°.

Chi, Kao, Chang, *J. Am. Chem. Soc.*, 1936, **58**, 1306.

Chu, Chou, *J. Am. Chem. Soc.*, 1947, **69**, 1257.

Peiminine

$C_{26}H_{41}O_3N.\frac{1}{2}H_2O$ MW, 424

Alkaloid of Chinese drug Pei-Mu from corms of *Fritillaria Roylei*, Stuart. Needles from EtOH. M.p. 135–7°.

B,HCl: cryst. M.p. 298°.

Monoacetyl: needles from Me_2CO. M.p. 174°.

Hydrochloride: prisms from EtOH–Et_2O.

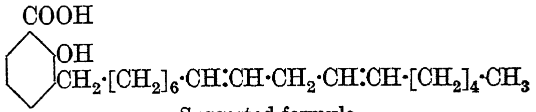
Oxime: cryst. M.p. 108° decomp.

Semicarbazone: cryst. M.p. 255–6°.

Phenylhydrazone hydrochloride: cryst. M.p. 266°.

Chu, Chou, *J. Am. Chem. Soc.*, 1941, **63**, 2937; 1947, **69**, 1257.

Pelandjauic Acid (*Pentaspadonic acid*)



Suggested formula

$C_{24}H_{36}O_3$ MW, 372

Obtained from Minjak Pelandjau, an oily exudation from wood of *Pentaspadon motleyi*, H. Cryst. M.p. 25–6°. $KMnO_4$ ⟶ azelaic and suberic acids.

Me ester: $C_{25}H_{38}O_3$. MW, 386. B.p. 225–35°/0·4 mm. D_4^{12} 0·9170. n_D^{12} 1·51477.

v. Romberg, v. Veen, *Chem. Abstracts*, 1930, **24**, 124.

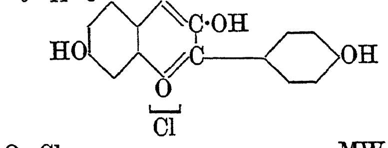
v. Rombergh, v. Veen, Smit, *Chem. Abstracts*, 1931, **25**, 937.

Backer, Haak, Grevenstok, *Rec. trav. chim.*, 1941, **60**, 678.

Pelargone.

See Di-*n*-octyl Ketone.

Pelargonenin chloride (*Pelargonidin* 5-β-*glucoside*)



$C_{21}H_{21}O_{10}Cl$ MW, 468·5

Scarlet-red needles. Sol. MeOH. Mod. sol. EtOH. Spar. sol. H_2O, HCl. Acid sols. ⟶ red col. with bluish fluor. Na_2CO_3.Aq. ⟶ violet-blue col.

Willstätter, Bolton, *Ann.*, 1916, **412**, 136.
Léon, Robertson, Robinson, Seshardi, *J. Chem. Soc.*, 1931, 2673.
Sondheimer, Kertesz, *J. Am. Chem. Soc.*, 1948, **70**, 3476.

Pelargonic Acid (*Nonanoic acid, nonylic acid, nonic acid, octane-*1*-carboxylic acid*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2{\cdot}COOH$

$C_9H_{18}O_2$ MW, 158

Oil. F.p. 12·24°. M.p. 15°. B.p. 255°/760 mm., 178·8°/64 mm., 121·2°/4 mm., 98·9°/1 mm. $D_4^{12\cdot5}$ 0·9109. $D_D^{17\cdot5}$ 0·9068. n_D 1·43057. $k = 1{\cdot}12 \times 10^{-5}$ at 25°. Spar. volatile in steam.

NH_4 salt: m.p. 115°.

Piperazinium salt: m.p. 95·1–96·2°.

Me ester: $C_{10}H_{20}O_2$. MW, 172. B.p. 213–14°/756 mm. $D^{17\cdot5}$ 0·8765.

Et ester: $C_{11}H_{22}O_2$. MW, 186. F.p. — 36·7°. B.p. 216–19° (227°). D_4^0 0·88156, D_4^{15} 0·86920.

2-*Chloroethyl ester*: b.p. 153°/20 mm. D_4^{11} 0·984.

2-*Iodoethyl ester*: b.p. 169°/15 mm.

2-*Diethylaminoethyl ester*: m.p. 131°.

n-*Propyl ester*: m.p. — 36°. B.p. 120–2°/20 mm. D_4^{25} 0·8540. n_D^{25} 1·4236.

n-*Butyl ester*: m.p. — 38°. B.p. 122–4°/20 mm. D_4^{25} 0·8520. n_D^{25} 1·4262.

tert.-*Butyl ester*: $C_{13}H_{26}O_2$. MW, 214. Liq. B.p. 242°.

n-*Amyl ester*: m.p. — 27°. B.p. 130–2°/20 mm. D_4^{25} 0·8506. n_D^{25} 1·4318.

Heptyl ester: $C_{16}H_{32}O_2$. MW, 256. F.p. — 15·54°. B.p. 210°/75 mm., 192·5°/30 mm. D_4^0 0·8745, D_4^{25} 0·8553. n_D^{20} 1·4350.

Vinyl ester: b.p. 133–133·5°/50 mm. D_4^{30} 0·8689. n_D^{30} 1·4447.

Allyl ester: b.p. 151°/50 mm. D_4^{30} 0·8702. n_D^{30} 1·4302.

2-*Chloroallyl ester*: b.p. 120°/5 mm. D_4^{30} 0·9719. n_D^{30} 1·4447.

Crotyl ester: b.p. 165°/50 mm. D_4^{30} 0·8709. n_D^{30} 1·4358.

p-*Chlorophenacyl ester*: m.p. 59°.

p-*Bromophenacyl ester*: m.p. 68·5°.

p-*Iodophenacyl ester*: m.p. 77°.

p-*Phenylphenacyl ester*: m.p. 70·8–71·3°.

Furfuryl ester: b.p. 151·5–153°/10 mm. D_4^{30} 0·9687. n_D^{30} 1·4563.

Chloride: $C_9H_{17}OCl$. MW, 176·5. F.p. — 60·5°. B.p. 215·35°/760 mm., 98°/15 mm. D_4^0 0·95901, D_4^{15} 0·94633.

Nitrile: $C_9H_{17}N$. MW, 139. F.p. — 34·2°. B.p. 224° (214–16°). D_4^0 0·83314, D_4^{15} 0·82207. Sol. EtOH, Et_2O. Insol. H_2O.

Amide: $C_9H_{19}ON$. MW, 157. Cryst. M.p. 98·9° (92–3°). Insol. cold H_2O.

Diethylamide: b.p. 167–9°/16 mm.

Isobutylamide: m.p. 37–8°. B.p. 162°/6 mm.

Anilide: cryst. from EtOH. M.p. 57·5°.

o-*Toluidide*: m.p. 73°.

p-*Toluidide*: cryst. from EtOH. M.p. 84°.

1-*Naphthylamide*: cryst. from EtOH. M.p. 91°.

Anhydride: f.p. 16° (5°). B.p. 207°/15 mm.

Eichler, *Ber.*, 1879, **12**, 1888.
Krafft, *Ber.*, 1882, **15**, 1691.
v. Braun, Sobecki, *Ber.*, 1911, **44**, 1469.
Rochussen, *Chem. Abstracts*, 1930, **24**, 1755.
Deffet, *Chem. Abstracts*, 1932, **26**, 352.
Moses, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 2101.

Pelargonic Aldehyde (*Nonanal, nonanoic aldehyde, nonaldehyde, nonylic aldehyde*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH_2{\cdot}CHO$

$C_9H_{18}O$ MW, 142

Found in mandarin and lemon-grass oil. B.p. 190–2°, 80–2°/13 mm. D^{15} 0·8277. n_D^{16} 1·42452. Polymerises readily with H_2SO_4. Oxidises slowly in air.

Trimer: m.p. 33·5°.

Di-Me acetal: b.p. 96–8°/15 mm. D_4^{18} 0·8733. $n_D^{18\cdot5}$ 1·4246.

Di-Et acetal: b.p. 130°/20 mm.

Oxime: leaflets from EtOH.Aq. M.p. 64°. Sol. most org. solvents. Volatile in steam.

Semicarbazone: cryst. from C_6H_6–pet. ether. or MeOH. M.p. 100°.

Thiosemicarbazone: m.p. 77°.

Phenylsemicarbazone: m.p. 131–2°.

o-*Tolylsemicarbazone*: m.p. 120–1°.

m-*Tolylsemicarbazone*: m.p. 124–5°.

p-*Tolylsemicarbazone*: m.p. 155–6°.

1-*Naphthylsemicarbazone*: m.p. 122–3°.

2-*Naphthylsemicarbazone*: m.p. 150–1°.

o-*Nitrobenzoylhydrazone*: m.p. 116·5°–117·5°.

m-*Nitrobenzoylhydrazone*: m.p. 87°.

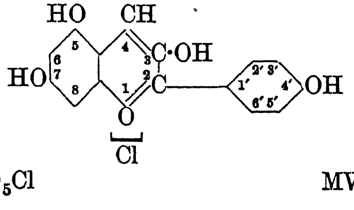
p-*Nitrobenzoylhydrazone*: m.p. 103–4°.

p-*Iodobenzoylhydrazone*: m.p. 135–6°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 100°.

Strain, *J. Am. Chem. Soc.*, 1935, **57**, 758.
Wagner, *Chem. Abstracts*, 1928, **22**, 3131.
Bagard, *Bull. soc. chim.*, 1907, **1**, 351.
Sabatier, Mailhe, *Compt. rend.*, 1912, **154**, 563; 1906, **158**, 986.
King, *J. Chem. Soc.*, 1938, 1826.
Scanlan, Swern, *J. Am. Chem. Soc.*, 1940, **62**, 2305.

Pelargonidin chloride



$C_{15}H_{11}O_5Cl$ MW, 306·5

Leaflets from MeOH–Et_2O, needles from MeOH–HCl. Does not melt below 350°. Sol. MeOH, EtOH. Mod. sol. H_2O, $CHCl_3$.

5-*Me ether*: $C_{16}H_{13}O_5Cl$. MW, 320·5. Cryst. Gives orange-red sol. in 0·5% HCl.

7-*Me ether*: $C_{16}H_{13}O_5Cl$. MW, 320·5. Solid. Gives orange-red sol. in 0·5% alc. HCl.

3 : 4′-*Di-Me ether*: $C_{17}H_{15}O_5Cl$. MW, 334·5. Crimson cryst. + H_2O. M.p. 221° decomp.

3-β-*Glucoside*: *See* Callistephin chloride.

4′-β-*Glucoside*: red plates. Decomp. at 184°. Sol. MeOH, EtOH. Spar. sol. cold H_2O, Me_2CO. *Picrate*: red prisms. Decomp. at 146–8°.

5-β-*Glucoside*: *see* Pelargonenin chloride.

7-β-*Glucoside*: scarlet needles. Sol. H_2O, EtOH, 0·5% HCl with deep red col. Na_2CO_3.Aq. ⟶ deep pink col. Alc. NaOH ⟶ violet-red col. *Picrate*: red plates. M.p. 180° decomp.

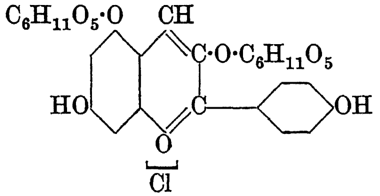
3 : 5-*Diglucoside*: *see* Pelargonin chloride.

5-*Benzoyl*: indigo plates. Sol. EtOH with violet col. Na_2CO_3.Aq. ⟶ violet col. 4′-*Tetra-acetylglucoside*: reddish-brown needles. Decomp. at 198°. AcOH ⟶ yellowish-red sol. with intense green fluor. Na_2CO_3.Aq. ⟶ bluish-pink sol. ⟶ blue with Me_2CO.

Picrate: reddish plates with golden reflex.

Willstätter, Burdict, *Ann.*, 1916, **412**, 163.
Robertson, Robinson, Sugiura, *J. Chem. Soc.*, 1928, 1533.
Léon, Robertson, Robinson, Seshadri, *J. Chem. Soc.*, 1931, 2673.
Nair, Robinson, *J. Chem. Soc.*, 1934, 1611.

Pelargonin chloride (*Pelargonidin 3 : 5-diglucoside*)



$C_{27}H_{31}O_{15}Cl$ MW, 630·5

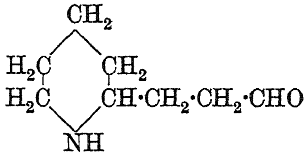
Fine red needles + $4H_2O$. M.p. anhyd. 180° decomp. $[\alpha]_D$ − 244°, $[\alpha]_{614}$ − 133° in 0·1% HCl. Sol. H_2O with orange-red sol. turning violet. Spar. sol. MeOH ⟶ red col. with green fluor. 20% HCl ⟶ pelargonidin + glucose.

Karrer, Widmer, *Helv. Chim. Acta*, 1927, **10**, 729.
Robinson, Todd, *J. Chem. Soc.*, 1932, 2488.

Note.—Robinson, Todd, (*loc. cit.*) have shown the identity of pelargonin, monardin and salvinin. Karrer and Meuron (*Helv. Chim. Acta*, 1932, **15**, 1214) have confirmed this. It is also possible that punicin may be identical with pelargonin.

Pelargonylacetic Acid.
See 2-Ketoundecylic Acid.

Pelletierine



$C_8H_{15}ON$ MW, 141

dl-.

Occurs in root-bark of pomegranate tree (*Runica granatum*). Colourless oil. B.p. 106°/21 mm. Readily absorbs O from air, becoming dark coloured and resinous. Sol. H_2O, Et_2O, $CHCl_3$. Alkaline in reaction.

B,HCl: needles from Me_2CO. M.p. 143–4°.

B,HBr: needles from Me_2CO. M.p. 140°.

Picrate: cryst. from EtOH. M.p. 150–1°.

N-*Acetyl*: b.p. 173–4°/18 mm. *B,HAuCl*$_4$: m.p. 95–6°.

Oxime: two forms. (i) From pet. ether. M.p. 96–7°; (ii) from Et_2O. M.p. 80°.

Semicarbazone hydrochloride: prisms from 50% AcOH. M.p. 188° decomp.

Hydrazone: b.p. 150°/20 mm.

Diethyl acetal: mobile liq. B.p. 101–2°/1·3 mm. *Picrolonate*: fine needles from EtOH. M.p. 162–3°.

N-*Carboethoxyl*: yellow oil. B.p. 173–4°/20 mm.

N-*Me*: $C_9H_{17}ON$. MW, 155. B.p. 98–102°/14 mm. Sol. H_2O. *B,HBr*: needles from Me_2CO or C_6H_6. M.p. 152°.

d-.

Sulphate: $[\alpha]_D^{18}$ + 5·86° in H_2O.

d-*Acid tartrate*: $[\alpha]_D^{20}$ + 21° in EtOH.

l-.

Sulphate: $[\alpha]_D^{18}$ − 5·89° in H_2O.

l-*Acid tartrate*: $[\alpha]_D^{20}$ − 20·94° in EtOH.

Hess, Eichel, *Ber.*, 1918, **51**, 743.
Hess, *Ber.*, 1919, **52**, 1005; 1917, **50**, 373.

Note.—Tanret (*Compt. rend.*, 1920, **170**, 1118) maintains that pelletierine occurs naturally in an optically active form, $[\alpha]_D$ − 31·1°.

Sulphate: m.p. 133°. $[\alpha]_D$ − 30·3°.

B,HCl: m.p. 145°. $[\alpha]_D$ − 41·2°.

B,HBr: m.p. 137°. $[\alpha]_D$ − 32·5°.

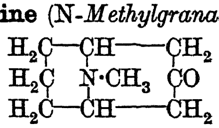
B,HNO$_3$: m.p. 82–5°. $[\alpha]_D$ − 34·8°.

N-*Acetyl*: b.p. 205–10°/40 mm. $[\alpha]_D$ + 32·6°.

Semicarbazone hydrochloride: m.p. 168–70°. $[\alpha]_D$ − 10·8°.

This active pelletierine is stated to be sensitive to acids and alkalis, and rapidly racemises.

ψ-Pelletierine (N-*Methylgranatonine*)



$C_9H_{15}ON$ MW, 153

Found in pomegranates. Prismatic plates from pet. ether. M.p. 48·5°. B.p. 246°/760 mm. Sol. EtOH, Et_2O, $CHCl_3$. Less sol. pet. ether. Sol. 2·5 parts H_2O. Strong base. CrO_3 ⟶ intense green col.

B,HAuCl₄: pale yellow cryst. from H_2O. M.p. 162° decomp. Sol. boiling H_2O.

Oxime: plates from Et_2O. M.p. 128–9°.

Methiodide: cryst. from EtOH.Aq. Does not melt below 280°.

N-*Oxide*: m.p. 160–2°. *B,HCl*: m.p. 224°.

Picrate: yellow needles from H_2O. M.p. 252–3° decomp.

Ciamician, Silber, *Ber.*, 1892, **25**, 1603; 1893, **26**, 156, 2738.
Piccinini, *Gazz. chim. ital.*, 1899, **29**, i, 408; 1899, **29**, ii, 312.
Hesse, *Ber.*, 1919, **52**, 1011.
Menzies, Robinson, *J. Chem. Soc.*, 1924, **125**, 2163.

Pellitorine (N-*Isobutylnona*-1 : 3-*diene*-1-*carboxyamide*)

$CH_3·[CH_2]_4·[CH:CH]_2·CONH·CH_2·CH(CH_3)_2$

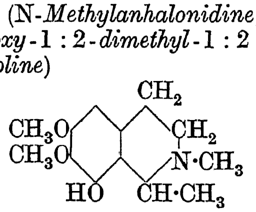
$C_{14}H_{25}ON$ MW, 223

Insecticidal constituent of *Anacyclus pyrethrum*, Pellitory. Needles from Skellysolve A. M.p. 73°.

Maleic anhydride add. comp.: m.p. 192°.

Gulland, Hopton, *J. Chem. Soc.*, 1930, 6.
Jacobson, *J. Am. Chem. Soc.*, 1949, **71**, 366.
Crombie, *Chemistry and Industry*, 1952, 1034.

Pellotine (N-*Methylanhalonidine*, 8-*hydroxy*-6 : 7-*dimethoxy*-1 : 2-*dimethyl*-1 : 2 : 3 : 4-*tetrahydroisoquinoline*)



$C_{13}H_{19}O_3N$ MW, 237

Found in *Anhalonium Williamsii*. Plates from EtOH. M.p. 111·5° (110°). Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$. Mod. sol. pet. ether. Spar. sol. H_2O. Aq. sol. reacts strongly alkaline. Conc. H_2SO_4 ⟶ yellow col. ⟶ reddish-violet with HNO_3. $FeCl_3$ ⟶ blue col. ⟶ green on warming. Slightly narcotic. Used as hypnotic.

B,HI: yellowish prisms. M.p. 125–30°. Sol. H_2O, EtOH. Insol. Et_2O.

B₂,H₂PtCl₆: m.p. 210–12°.

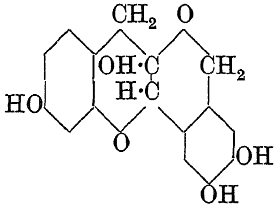
B,HAuCl₄: orange cryst. M.p. 147–8° decomp.

Methiodide: plates or prisms + H_2O from MeOH. M.p. 199°. *Me ether*: prisms from H_2O. M.p. 225°. *Et ether*: m.p. 185–6°. *Benzyl ether*: m.p. 193–5°.

Picrate: yellow prisms from H_2O. M.p. 172–3°.

Hefter, *Ber.*, 1894, **27**, 2975; 1901, **34**, 3004.
Späth, *Monatsh.*, 1921, **42**, 97; *Ber.*, 1932, **65**, 1778.
Späth, Boschan, *Monatsh.*, 1933, **63**, 146.
Späth, Becke, *Ber.*, 1934, **67**, 266.
Späth, Kesztler, *Ber.*, 1936, **69**, 755.

Peltogynol



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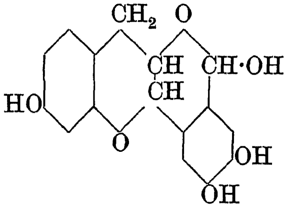


$C_{16}H_{14}O_6$ MW, 302

Found in heart-wood of *Peltogyne porphyrocardia*. Prisms from H_2O. Becomes pink at 200°, dark red at 240° with decomp. and softening. Sol. MeOH, EtOH. Mod. sol. Me_2CO. Spar. sol. cold H_2O, Et_2O, $CHCl_3$, C_6H_6. Aq.NaOH ⟶ blue sol. ⟶ bright red on standing. Alc. $FeCl_3$ ⟶ blue col. Hot HNO_3 ⟶ styphnic acid.

Tri-Me ether: $C_{19}H_{20}O_6$. MW, 344. Long needles or plates from AcOEt. M.p. 198°. $[\alpha]_D$ + 250° in $CHCl_3$.

Tetra-Me ether: $C_{20}H_{22}O_6$. MW, 358. Plates. M.p. 175°. $[\alpha]_D^{20}$ + 264° in $CHCl_3$.

Tetra-acetyl: needles from AcOH or C_6H_6–EtOH. M.p. 173°. Sol. AcOEt, $CHCl_3$, C_6H_6. Spar. sol. H_2O, MeOH, EtOH, Et_2O, pet. ether. $[\alpha]_D^{20}$ + 125° in $CHCl_3$.

Tetrabenzoyl: plates from AcOH. M.p. 244°. Very sol. $CHCl_3$. Mod. sol. AcOEt.

Tetra-anisoyl: plates from AcOH. M.p. 218° decomp. Sol. C_6H_6. Mod. sol. Me_2CO. Spar. sol. MeOH, Et_2O.

Robinson, Robinson, *J. Chem. Soc.*, 1935, 744.

Penaldic-F Acid (2-*Pentenylpenaldic acid*)

$CH_3·CH_2·CH:CH·CH_2·CO·NH·CH<{COOH \atop CHO}$

$C_9H_{13}O_4N$ MW, 199

Me ester: $C_{10}H_{15}O_4N$. MW, 213. 2 : 4-*Dinitrophenylhydrazone*: needles from EtOH. M.p. 146°.

Et ester: $C_{11}H_{17}O_4N$. MW, 227. Oil. 2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH. M.p. 159–60°.

Benzyl ester: $C_{16}H_{19}O_4N$. MW, 289. Cryst. from C_6H_6–light petroleum. M.p. 88°.

Di-Et acetal: $C_{13}H_{23}O_5N$. MW, 273. Cryst. + $1H_2O$ from EtOH.Aq. M.p. 79°.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 473.

Penaldic-G Acid (*Benzylpenaldic acid*)

$C_6H_5{\cdot}CH_2{\cdot}CO{\cdot}NH{\cdot}CH{<}^{COOH}_{CHO}$

$C_{11}H_{11}O_4N$ MW, 221

dl-.

Me ester: $C_{12}H_{13}O_4N$. MW, 235. *Enol benzoate*: cryst. from EtOH. M.p. 162–5°. *Enol* p-*nitrobenzoate*: cryst. from EtOH. M.p. 169·5–170°. *Enol toluene*-p-*sulphonate*: cryst. from EtOH. M.p. 147–8°. *Anil*: needles. M.p. 162–3°.

Et ester: $C_{13}H_{15}O_4N$. MW, 249. Yellow oil. *Enol acetate*: cryst. from Et_2O. M.p. 96–7°.

Anil: needles. M.p. 160°. 2 : 4-*Dinitrophenylhydrazone*: m.p. 188–9°.

Benzyl ester: $C_{18}H_{17}O_4N$. MW, 311. Needles from cyclohexane. M.p. 96–7°. $FeCl_3$ ⟶ grayish-purple col. 2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH. M.p. 178–9°.

Di-Me acetal: $C_{13}H_{17}O_5N$. MW, 267. Cryst. from AcOEt–light petroleum. M.p. 109–11°. *Me ester*: $C_{14}H_{19}O_5N$. MW, 281. Brown oil. *Benzylamide*: needles from MeOH. M.p. 163–5°.

Di-Et acetal: $C_{15}H_{21}O_5N$. MW, 295. Leaflets from $CHCl_3$–light petroleum. M.p. 110° decomp. *Me ester*: $C_{16}H_{23}O_5N$. MW, 309. Cryst. from MeOH.Aq. M.p. 77–9° (106°). *Et ester*: $C_{17}H_{25}O_5N$. MW, 323. Amber oil. *Amide*: $C_{15}H_{22}O_4N_2$. MW, 294. Needles. M.p. 170–1°. *Anilide*: cryst. from light petroleum. M.p. 69–70°. *Hydrazide*: needles from EtOH. M.p. 164–5°.

Di-butyl acetal: $C_{19}H_{29}O_5N$. MW, 351. Cryst. from EtOH. M.p. 106°. *Me ester*: $C_{20}H_{31}O_5N$. MW, 365. M.p. 28–30°. B.p. 151–2°/0·022 mm. n_D^{25} 1·4900.

Di-Et mercaptal: $C_{15}H_{21}O_3NS_2$. MW, 327. Needles from EtOH.Aq. M.p. 131–2°.

Benzylamide: *semicarbazone*, prisms from EtOH.Aq. M.p. 216–17°.

Dimedone deriv.: cryst. from MeOH.Aq. M.p. 151–8°.

2 : 4-*Dinitrophenylhydrazone*: cryst. from MeOH. M.p. 180–1°. *Benzylamide*: cryst. M.p. 242–3°.

d-.

Di-Me acetal: cryst. from $CHCl_3$. M.p. 136°. $[\alpha]_D^{25}$ + 34·8° in $CHCl_3$. *Brucine salt*: cryst. + $6\frac{1}{2}H_2O$ from H_2O. M.p. 80–100°. $[\alpha]_D$ − 26·3° in EtOH. *Me ester*: cryst. from hexane–AcOEt. M.p. 49–50°. $[\alpha]_D^{23}$ + 20° in $CHCl_3$, − 10° in EtOH.

l-.

Di-Me acetal: cryst. from $CHCl_3$. M.p. 130°. $[\alpha]_D^{25}$ − 17·6° in $CHCl_3$.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 473.

Penaldic-K Acid (*Heptylpenaldic acid*)

$CH_3{\cdot}[CH_2]_5{\cdot}CH_2{\cdot}CO{\cdot}NH{\cdot}CH{<}^{COOH}_{CHO}$

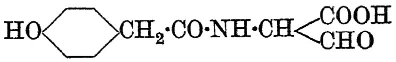
$C_{11}H_{19}O_4N$ MW, 229

Di-Et acetal: $C_{15}H_{29}O_4N$. MW, 303. Cryst. from EtOH.Aq. M.p. 72–3°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 187–8°.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 473.

Penaldic-X Acid (p-*Hydroxybenzylpenaldic acid*)



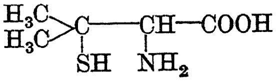
$C_{11}H_{11}O_5N$ MW, 237

Me ester: $C_{12}H_{13}O_5N$. MW, 251. Yellow oil. $FeCl_3$ ⟶ faint purple col. 2 : 4-*Dinitrophenylhydrazone*: cryst. M.p. 223–4°.

Di-Et acetal: $C_{15}H_{21}O_6N$. MW, 311. Cryst. from Et_2O–C_6H_6. M.p. 70–5° decomp. O-*Acetyl*: cryst. from EtOH.Aq. M.p. 118–19°.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 473.

Penicillamine (2-*Mercapto*-1-*aminoisovaleric acid*, β-*mercaptovaline*, ββ-*dimethylcysteine*)



$C_5H_{11}O_2NS$ MW, 149

d-.

Characteristic degradation product of the penicillins. pK_1 (acid; carboxyl group), 1·8; pK_2 (acid; mercapto group), 10·4; pK (base; amino group), 7·9. Me_2CO ⟶ 2 : 2 : 5 : 5-tetramethylthiazolidine-4-carboxylic acid, m.p. 198°. Air ⟶ disulphide.

B,HCl: cryst. M.p. 160–5°. $[\alpha]_D$ + 1° in H_2O, − 55° in *N*/NaOH. $FeCl_3$ ⟶ transient deep blue col. Na nitroprusside ⟶ purple col. Br ⟶ penicillaminic acid, $(CH_3)_2C(SO_3H){\cdot}CH(NH_2){\cdot}COOH$. Prisms. M.p. above 325°.

Me ester: oil. *B,HCl*: needles. M.p. 180–2°. $[\alpha]_D$ − 7·5° in H_2O. *Isopropylidene deriv.*:

m.p. 18–20°. B.p. 59°/0·1 mm. $[\alpha]^{25}_{5461}$ + 116° in EtOH.

Et ester: oil. *B,HCl*: cryst. M.p. 161–2°.

N-*Formyl*: cryst. from H_2O or C_6H_6–pet. ether. M.p. 179–80°. $[\alpha]^{22}_{D}$ + 62° in H_2O.

N-*Acetyl*: cryst. from H_2O. M.p. 189–90°. $[\alpha]^{25}_{D}$ + 18° in 50% EtOH.Aq.

N-*Phenylacetyl*: m.p. 139–40°. $[\alpha]_D$ + 40°.

N-*Carbobenzyloxy*: m.p. 142–3°.

N-*Methyl*: *B,HCl*: m.p. 160–70°.

S-*Benzyl*: m.p. 197–8° decomp. $[\alpha]^{17}_{D}$ + 90° in *N*/NaOH. *B,HCl*: cryst. + $1H_2O$. $[\alpha]_D$ − 75° in *N*/HCl. *Me ester*: *B,HCl*, m.p. 147–8°. *Et ester*: *B,HCl*: m.p. 145–6°. $[\alpha]_D$ − 102°.

Disulphide: $C_{10}H_{20}O_4N_2S_2$. MW, 296. Cryst. from EtOH.Aq. M.p. 204–5° decomp. $[\alpha]^{23}_{D}$ + 27° in *N*/HCl, − 82° in *N*/NaOH, + 118° in H_2O.

l-.

Cryst. M.p. 190–4° decomp. $[\alpha]_D$ + 63° in *N*/NaOH.

N-*Formyl*: cryst. from H_2O. M.p. 179–80°. $[\alpha]^{22}_{D}$ − 54°.

S-*Benzyl*: m.p. 197–8° decomp. $[\alpha]^{17}_{D}$ − 90° in *N*/NaOH. *B,HCl*: cryst. + $1H_2O$. $[\alpha]_D$ + 75° in *N*/HCl.

Disulphide: cryst. from EtOH.Aq. M.p. 207° decomp. $[\alpha]^{22}_{D}$ − 26° in *N*/HCl, + 76° in *N*/NaOH. *Diformyl*: cryst. from Me_2CO–C_6H_6. M.p. 187° decomp. $[\alpha]^{27}_{D}$ − 29° in Py.

dl-.

Cryst. M.p. 201° decomp.

B,HCl: m.p. 145–8° decomp.

Me ester: oil. *B,HCl*: cryst. from *tert.*-butyl alcohol. M.p. 173–5°. *Isopropylidene deriv.*: m.p. 48–49°.

Et ester: oil. *B,HCl*: m.p. 150–1°. *Isopropylidene deriv.*: b.p. 42·5/0·01 mm.

Propyl ester: oil. *B,HCl*: m.p. 137–8°.

Isopropyl ester: oil. *B,HCl*: m.p. 162–3°.

Butyl ester: oil. *B,HCl*: m.p. 125°.

Isobutyl ester: oil. *B,HCl*: m.p. 131–2°.

Isoamyl ester: oil. *B,HCl*: m.p. 130–1°.

Benzyl ester: oil. *B,HCl*: m.p. 159–60°.

N-*Formyl*: cryst. from H_2O. M.p. 157–8°.

N-*Acetyl*: m.p. 183°.

N-*Benzoyl*: m.p. 135–6°.

N-*Benzyl*: cryst. M.p. 194–6°. *B,HCl*: m.p. 182–4°.

S-*Benzyl*: cryst. from H_2O. M.p. 202° decomp. N-*Formyl*: cryst. from EtOH. M.p. 158–9°. N-*Acetyl*: m.p. 177–8°.

Disulphide: two forms. (i) M.p. 206°. (ii) M.p. 181–3°.

Clarke, Johnson, Robinson, *The Chemistry of Penicillin*, Princeton University Press, 1949.

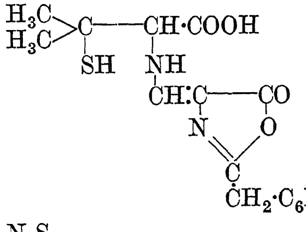
Süs, *Ann.*, 1948, **559**, 92.

Tatsuoko, Mujamoto, Shiu, Tamura, *Journal of Penicillin* (*Japan*), 1947, **1**, 382.

Penicillaminic Acid.

See under Penicillamine.

Penicillenic-G Acid (*Benzylpenicillenic acid*)



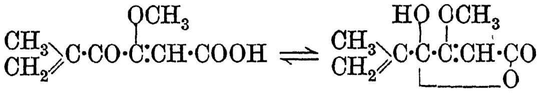
$C_{16}H_{18}O_4N_2S$ MW, 334

d-.

Me ester: light yellow oil. $[\alpha]^{28}_{D}$ + 40° in C_6H_6. Shows ultra-violet absorption max. at 317·5 mμ (ε, 28,000). Heat ⟶ methyl penillonate-G. S-*Benzyl*: cryst. from Et_2O. M.p. 54–5°. "*Hydrochloride*": m.p. 65–70° decomp. "*Hydrobromide*": m.p. 110–15° decomp.

Cornforth, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 833.

Penicillic Acid



$C_8H_{10}O_4$ MW, 170

Metabolic product of *Penicillium puberulum* Bainier, and *Penicillium cyclopium* Westling. Rhombic or hexagonal plates + $1H_2O$ from H_2O. M.p. 64–5°, anhyd. 87°. Sol. to 2% in cold H_2O. Readily sol. hot H_2O, EtOH, Et_2O, C_6H_6, $CHCl_3$. Insol. pet. ether. NH_4OH ⟶ reddish purple col.

Acetyl: prisms from pet. ether. M.p. 72°.

Dimedone deriv.: $C_8H_{10}O_4$, $C_8H_{12}O_2$. Cryst. from EtOH.Aq. M.p. 201–3°.

Dihydro-comp.: needles + $1H_2O$ from H_2O. M.p. 62–4°, anhyd. 83–5°.

Dibromide: needles from CCl_4. M.p. 154–5°.

Birkinshaw, Oxford, Raistrick, *Biochem. J.*, 1936, **30**, 394.

Oxford, Raistrick, Smith, *Chemistry and Industry*, 1942, 22.

Raphael, *J. Chem. Soc.*, 1948, 1508.

Penicillin-I.

See Penicillin-F.

Penicillin-II.

See Penicillin-G.

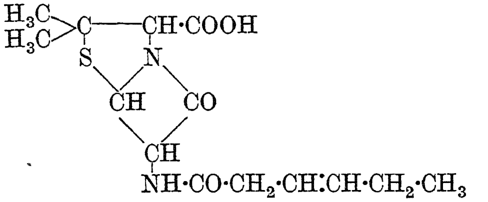
Penicillin-III.

See Penicillin-X.

Penicillin-IV.

See Penicillin-K.

Penicillin-F (*Pencillin-I, 2-pentenylpenicillin*)



$C_{14}H_{20}O_4N_2S$ MW, 304

Powerful antibiotic from members of the *Penicillium* group. Sol. Et_2O, $CHCl_3$, alcohols, ketones, esters. Spar. sol. CCl_4, hydrocarbons. pK 2·8 in H_2O at 5°, 2·87 at 25°. Unstable in H_2O outside pH range 5–8. Cold acid ⟶ penillic-F acid. Hot acid ⟶ penicillamine + penilloaldehyde-F. Alkalis ⟶ penicilloic-F acid. MeOH ⟶ α-methyl penicilloate-F. $HgCl_2$ ⟶ penicillamine + penilloaldehyde-F. Benzylamine ⟶ α-benzylamide of penicilloic-F acid. Inactivated by penicillinase ⟶ penicilloic-F acid.

Na salt: colourless needles + $3H_2O$ from AcOEt.Aq. M.p. 204–5° decomp. $[\alpha]_D^{23}$ + 316° in H_2O. Also forms cryst. sesquihydrate. Anhyd. exists in two cryst. forms. Antibiotic activity: 1,490 Penicillin units per mg.

Ba salt: sol. H_2O. $[\alpha]_D^{22}$ + 228° in H_2O.

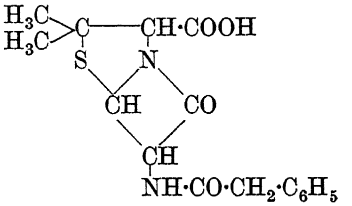
Rb salt: needles from Et_2O-*n*-butyl alcohol.

Me ester: oil. $[\alpha]_D^{25}$ + 186° in MeOH. Antibiotic activity: 15 Penicillin units per mg. Shows high *in vivo* activity against *Streptomyces pyogenes* in mice. $HgCl_2$ ⟶ penicillamine Me ester + penilloaldehyde-F.

Boon, Carrington, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 77.

Abraham, Baker, Boon, Calam, Carrington, Chain, Florey, Freeman, Robinson, Saunders, *ibid.*, p. 10.

Penicillin-G (*Penicillin-II, benzylpenicillin, parasiticin*)



$C_{16}H_{18}O_4N_2S$ MW, 334

Potent antibiotic elaborated by strains of the moulds *Penicillium* and *Aspergillus*. Amorphous white unstable powder. $[\alpha]_D$ + 282° in EtOH. Antibiotic activity: 1,250 Penicillin units per mg. Usually isolated as Na salt. Cold aq. acids ⟶ penillic-G acid. Hot aq. acids ⟶ penicillamine + penilloaldehyde-G. Alkaline inactivation ⟶ penicilloic-G acid. Hot alkalis ⟶ α-phenyl-levulinic acid. Alcohols ⟶ α-alkylpenicilloates-G. Benzylamine ⟶ benzylamine salt of penicilloic-G acid α-benzylamide. Se ⟶ phenylacetamide. Inactivated by penicillinase. Stable when pure and dry.

Na salt: needles from MeOH–AcOEt or H_2O–*n*-butyl alcohol. M.p. 215° decomp. $[\alpha]_D^{24\cdot8}$ + 301° in H_2O. $[\alpha]_{5463}$ + 368° in H_2O. Shows ultra-violet absorption maxima at 252 (ε, 300), 257·5 (ε, 240) and 264 mμ (ε, 180). Sol. H_2O, MeOH. Less sol. EtOH. Spar. sol. isopropyl alcohol, *tert.*-butyl alcohol, AcOEt, Py. Sol. to 0·081% in *n*-butyl alcohol at 0°, to 0·006% in Me_2CO at 0°, to 0·0024% in methyl ethyl ketone at 0°, and to 0·015% in dioxan at 14°. (*N.B.* These solubilities refer to solubilities of the pure salt in dry solvents. Crude preparations are very much more soluble, and the pure salt is more soluble in damp solvents than indicated.) Antibiotic activity: 2,100 Penicillin units per mg. Inactivated by penicillinase and *p*-hydroxyphenylacetic acid.

K-salt: fine needles from H_2O–*n*-butyl alcohol. M.p. 214–17° decomp. $[\alpha]_D^{22}$ + 285° in H_2O.

Ca salt: amorphous. Sol. Me_2CO, AcOEt, $CHCl_3$. Antibiotic activity: 1,400 Penicillin units per mg.

Ba salt: amorphous. Sol. Me_2CO, AcOEt, $CHCl_3$.

NH_4 salt: blades from 80% Me_2CO–dry Me_2CO. M.p. 137–8° decomp. $[\alpha]_D^{24}$ + 299° in H_2O.

Triethylammonium salt: clusters of cryst. from chlorobenzene. M.p. 145–7° decomp. $[\alpha]_D^{23}$ + 236°.

Benzylammonium salt: needles. M.p. 100°. $[\alpha]_D$ + 253° in MeOH. In Et_2O or MeOH ⟶ α-benzylamide of benzylpenicilloic acid.

Procaine salt: cryst. M.p. 129–30°. $[\alpha]_D^{25}$ + 173° in 50% Me_2CO.Aq. Antibiotic activity: 1,020 Penicillin units per mg.

N-Me-piperidinium salt: m.p. 135–6° (rapid heat.). Sol. H_2O to sol. of pH 4·5. $[\alpha]_D^{25}$ + 244°.

N-Et-piperidinium salt: cryst. from $CHCl_3$–Me_2CO. M.p. 167–8° decomp. (rapid heat). Sol. H_2O to sol. of pH 5·3. $[\alpha]_D^{25}$ + 238°. Antibiotic activity: 1,287 Penicillin units per mg.

N-Et-morpholinium salt: cryst. M.p. 140–1° (rapid heat). Sol. H_2O to sol. of pH 4·8. $[\alpha]_D^{25}$ + 238°.

Me ester: needles from AcOEt–hexane or CCl_4. M.p. 97–8°. $[\alpha]_D$ + 286° (+ 179°) in $CHCl_3$, + 328° in MeOH. Sublimes at 130–35°/1–3 × 10^{-6} mm.

Benzyl ester: oil. $[\alpha]_D^{24}$ + 213°. Sol. AcOEt. Insol. pentane. Antibiotic activity: 15–16 Penicillin units per mg.

p-*Iodobenzyl ester*: white amorphous solid. $[\alpha]_D^{21}$ + 182° in MeOH.

2-*Dimethylaminoethyl ester*: cryst. M.p. 138–9° decomp. $[\alpha]_D^{21} + 202°$ in EtOH. Rapidly hyd. by H_2O.

Anhydride: amorph. $[\alpha]_D^{22} + 175°$ in $CHCl_3$.

Amide: cryst. + $3H_2O$. M.p. 65°, anhyd., 161°. Potent antibiotic. Not inactivated by penicillinase.

Peck, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, pp. 52, 144.
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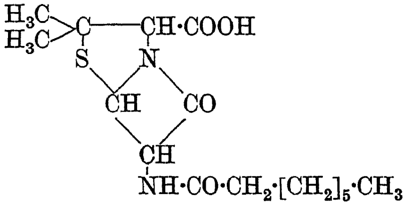
Carpenter, *J. Am. Chem. Soc.*, 1948, **70**, 2964.
Cooper, Binkley, *ibid.*, 3966.
Salivar, Hedger, Brown, *ibid.*, 1287.
du Vigneaud *et al.*, *J. Biol. Chem.*, 1948, **175**, 721; 1948, **176**, 893, 915; *Science*, 1946, **104**, 431.
Süs, *Ann.*, 1951, **571**, 201.

Penicillin-K (*Penicillin-IV*, n-*heptylpenicillin*)



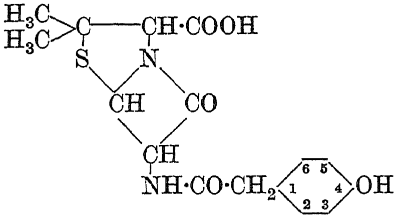
$C_{16}H_{26}O_4N_2S$ MW, 342

Powerful antibiotic elaborated by *Penicillium* strains. Cold acids ⟶ penillic-K acid. Hot $6N$/HCl ⟶ caprylic acid. $HgCl_2$ ⟶ penicillamine + penilloaldehyde-K. MeOH ⟶ α-methyl penicilloate-K. Benzylamine ⟶ α-benzylamide of penicilloic-K acid. pK 3·2.

Na salt: needles + $1H_2O$ from AcOEt–Me_2CO–H_2O. Antibiotic activity: 2,400 Penicillin units per mg.

McCorquodale, Risser, Philip, Touster, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 98.

Penicillin-X (*Penicillin-III*, p-*hydroxybenzylpenicillin*)



$C_{16}H_{18}O_5N_2S$ MW, 350

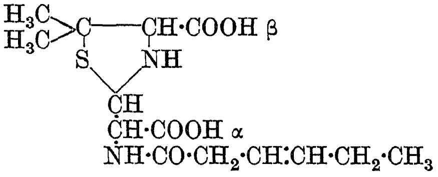
Potent antibiotic from *Penicillium* strains.

Na salt: rosettes from H_2O–*n*-butyl alcohol or 80% Me_2CO. Shows ultra-violet absorption max. at 275 mμ (ε 1,405). Cold min. acids ⟶ penillic-X acid. Benzylamine ⟶ α-benzylamide of penicilloic-X acid. Hot 10% HCl ⟶ *p*-hydroxyphenylacetic acid.

3 : 5-*Diiodo*: *di-Na salt*: colourless needles + $2Me_2CO$ from Me_2CO.Aq. Antibiotic activity: 1,715 Penicillin units per mg.

McCorquodale, Stodola, Wachtel, Coghill, *The Chemistry of Penicillin*, Princeton University Press, 1949, pp. 95, 680.

Penicilloic-F Acid (2-*Pentenylpenicilloic acid*)



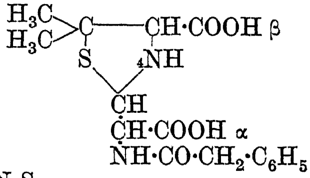
$C_{14}H_{22}O_5N_2S$ MW, 330

α-*Me ester*: pale yellow amorphous solid. $[\alpha]_D^{23} + 92°$ in H_2O. *Na salt*: $[\alpha]_D^{23} + 159°$ in H_2O.

α-*Benzylamide*: *benzylamine salt*, cryst. M.p. 130–1°.

Mozingo, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 535.

Penicilloic-G Acid (*Benzylpenicilloic acid*)



$C_{16}H_{20}O_5N_2S$ MW, 352

d-.

Four stereoisomeric forms known.

I. α-.

α-*Me ester*: cryst. from dibutyl ether–AcOEt. M.p. 125–7°.

β-*Me ester*: glass. *Benzylamine salt*: cryst. from MeOH–Et_2O. M.p. 138–40°.
α-*Et ester*: amorphous. *Benzylamine salt*: cryst. M.p. 140–1°.
α-*Benzyl ester*: *benzylamine salt*: m.p. 162–3°.
Di-Me ester: cryst. M.p. 87–9°.
α-*Et* β-*Me ester*: cryst. from Et_2O. M.p. 85–6°.

II. β-.

α-*Me ester*: N^4-*benzoyl*, cryst. from MeOH–Et_2O–light petroleum. M.p. 235–6°.
α-*Et ester*: N^4-*benzoyl*, cryst. M.p. 233–5°.
Di-Me ester: needles. M.p. 113–14°.
α-*Et* β-*Me ester*: needles from Et_2O–light petroleum. M.p. 100–1°. $[\alpha]_D^{24}$ + 21·7° in EtOH. $[\alpha]_D^{28}$ + 48° in AcOH.

III. γ-.

Amorphous. M.p. 115–20° decomp.
α-*Me ester*: cryst. from MeOH. M.p. 161–3° decomp. $[\alpha]_D^{28}$ + 135·7°.
β-*Me ester*: unstable white amorph. solid. M.p. 50–60°.
α-*Et ester*: needles from EtOH. M.p. 150° decomp. $[\alpha]_D$ + 128° in EtOH. pK, 4·10.
α-*Benzyl ester*: needles from $CHCl_3$. M.p. 164–5°. $[\alpha]_D^{28}$ + 125·5° in EtOH.
Di-Me ester: needles from $CHCl_3$–Et_2O. M.p. 107–9°. $[\alpha]_D^{28}$ + 100° in EtOH.
α-*Et* β-*Me ester*: cryst. from Et_2O. M.p. 87–8°. $[\alpha]_D$ + 103° in EtOH.
α-*Hydrazide*: cryst. from EtOH.Aq. M.p. 154°, resolidifies and then melts at 188°.
α-*Benzylamide*: needles from Me_2CO.Aq. M.p. 119–21°.
Di-hydrazide: cryst. M.p. 172–3°.

IV. δ-.

Amorph. Sinters at 105°, m.p. 115°. $[\alpha]_D^{24}$ − 23·1° in EtOH.
β-*Me ester*: white powder. $[\alpha]_D^{25}$ + 4° ⟶ + 24° in MeOH.
Di-Me ester: cryst. from $CHCl_3$–Et_2O. M.p. 111–14°. $[\alpha]_D^{23}$ − 40° in MeOH.

l-. γ-.

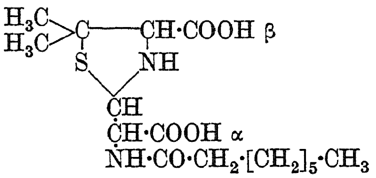
Amorph. M.p. 110–15° decomp. $[\alpha]_D$ − 102·9° in EtOH.
α-*Me ester*: cryst. M.p. 158–9°. $[\alpha]_D^{28}$ − 129·1° in EtOH.
β-*Me ester*: α-*benzylamide*, m.p. 100–25° decomp.

dl-. γ-.

Amorph. M.p. 130–40°.

Mozingo, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 535.

Penicilloic-K Acid (*Heptylpenicilloic acid*)

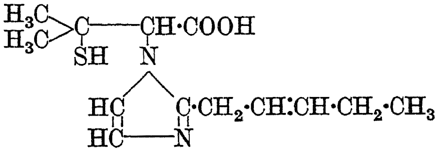


$C_{16}H_{28}O_5N_2S$ MW, 360

α-*Benzylamide*: cryst. from EtOH.Aq. M.p. 80–90°. *Benzylamine salt*: cryst. + $\frac{1}{2}H_2O$. M.p. 125–6°.
α-*Me ester*: *benzylamine salt*, two forms. α-, M.p. 74–6°, resolidifying to β-form, m.p. 80–3°.

Mozingo, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 535.

Penillamine-F (2-*Pentenylpenillamine*)

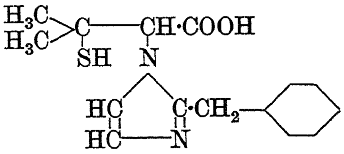


$C_{13}H_{20}O_2N_2S$ MW, 268

d-.
B,HCl: plates from MeOH–Et_2O. M.p. 175°. $[\alpha]_{5461}^{20}$ − 88° in H_2O.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillamine-G (*Benzylpenillamine*)



$C_{15}H_{18}O_2N_2S$ MW, 290

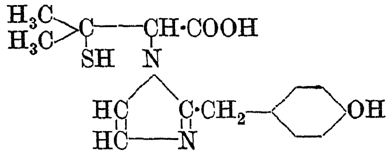
d-.
B,HCl: needles from Me_2CO–Et_2O. M.p. 173°. $[\alpha]_D^{20}$ − 66° in H_2O. Shows ultra-violet absorption max. at 240 (ε 1,200) and 230 mμ (ε 3,800). pK (acid) 2·81, 10·35; pK (base) 6·98.
Picrate: needles or dendritic clusters. M.p. 156–7°.

l-.
B,HCl: prismatic needles from Me_2CO–Et_2O. M.p. 173–4° decomp. $[\alpha]_D^{18}$ + 65° in H_2O.

dl-.
B,HCl: prisms. M.p. 193°.
Picrate: m.p. 159–60°.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillamine-X (p-*Hydroxybenzylpenillamine*)



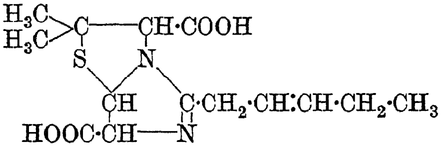
$C_{15}H_{18}O_3N_2S$ MW, 306

dl-.

B,HCl: prismatic needles from MeOH–Me_2CO–Et_2O. M.p. 175–6°.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillic-F Acid (2-*Pentenylpenillic acid*)



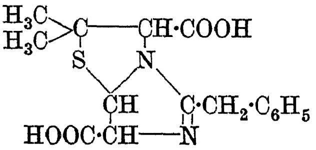
$C_{14}H_{20}O_4N_2S$ MW, 312

d-.

Acid inactivation product of penicillin-F. Orthorhombic plates from H_2O. M.p. 175° (effervescence). $[\alpha]_D^{16·5}$ + 527°, $[\alpha]_D^{19}$ + 507°, $[\alpha]_{5461}$ + 600°. Shows ultra-violet absorption max. at 230 mμ (ε 6,200) in 1*N*/HCl; at 238 mμ (ε 6,000) at pH 3. Hyd. ⟶ penicillamine + penilloaldehyde-F. $HgCl_2$ ⟶ penillamine-F. Hot H_2O ⟶ penilloic-F acid.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillic-G Acid (*Benzylpenillic acid*)



$C_{16}H_{18}O_4N_2S$ MW, 334

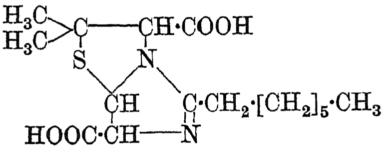
d-.

Acid inactivation product of penicillin-G. Needles, hexagonal plates or long hexagons from H_2O or MeOH.Aq. M.p. 193–4° decomp. (179–80° decomp., 186–7° decomp.). $[\alpha]_D^{16·5}$ + 500 ± 10°, $[\alpha]_D^{22}$ + 548° in 0·1*N*/NaOH, $[\alpha]_{5463}$ + 678° in 0·1*N*/NaOH, $[\alpha]_{5693}$ + 536° in 0·1*N*/NaOH. $[\alpha]_D$ + 545° in NH_3.Aq. $[\alpha]_D^{23}$ + 538° in H_2O, $[\alpha]_D^{23}$ + 465° in MeOH. Shows ultra-violet absorption max. in H_2O at 280 (ε 2,000), 240 (ε 6,500) and 210 mμ (ε 10,000). pK 3·2–3·4 and 7·7. Hyd. ⟶ penicillamine + penilloaldehyde-G. Hot H_2O ⟶ penilloic-G acid.

Di-Me ester: hexagonal cryst. or prisms. M.p. 135–6° (118–21°, 126–7°). $[\alpha]_D^{23}$ + 411° in MeOH. $[\alpha]_{5463}$ + 50·6° in MeOH.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillic-K Acid (*Heptylpenillic acid*)



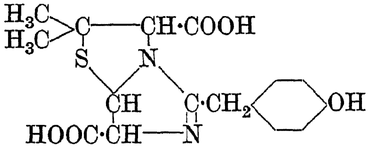
$C_{16}H_{26}O_4N_2S$ MW, 342

d-.

Hexagonal plates from H_2O. M.p. 176°. $[\alpha]_D^{22}$ + 490°. Shows ultra-violet absorption max. at 238 mμ (ε 5,900).

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penillic-X Acid (p-*Hydroxybenzylpenillic acid*)



$C_{16}H_{18}O_5N_2S$ MW, 350

d-.

Diamond-shaped cryst. from H_2O. M.p. 217°. $[\alpha]_D^{17·5}$ + 478° (Na salt in H_2O). Shows ultra-violet absorption max. at 225–227 mμ (ε 8,000). $FeCl_3$ ⟶ yellow col. Hot HCl ⟶ *p*-hydrophenylacetic acid.

O-*Carbobenzyloxy deriv.*: needles from EtOH. M.p. 186°.

Cook, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 106.

Penilloaldehyde-F (2-*Pentenylpenilloaldehyde*, 3-*hexenoamidoacetaldehyde*)

$CH_3·CH_2·CH{:}CH·CH_2·CO·NH·CH_2·CHO$

$C_8H_{13}O_2N$ MW, 155

2 : 4-*Dinitrophenylhydrazone*: yellow needles from EtOH. M.p. 187–8°.

Abraham, Chain, Baker, Robinson, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 34.

Penilloaldehyde-G (*Benzylpenilloaldehyde*, *phenylacetamidoacetaldehyde*)

$C_6H_5·CH_2·CO·NH·CH_2·CHO$

$C_{10}H_{11}O_2N$ MW, 177

Flakes. M.p. 113·5–115·5°. Hyd. ⟶ phenylacetic acid.

Di-Et acetal: cryst. from C_6H_6–light petroleum. M.p. 40–1°.

Di-butyl acetal: cryst. from light petroleum. M.p. 42°.

2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 198°.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 482.

Penilloaldehyde-K (*Heptylpenilloaldehyde, octanoamidoacetaldehyde*)

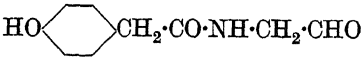
$CH_3·[CH_2]_5·CH_2·CO·NH·CH_2·CHO$

$C_{10}H_{19}O_2N$ MW, 185

Di-Et acetal: oil. B.p. 100–1°/2mm. n_D^{25} 1·4484.
2 : 4-*Dinitrophenylhydrazone*: needles from $CHCl_3$–light petroleum. M.p. 171–3°.

McCorquodale, Risser, Philip, Touster, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 101.

Penilloaldehyde-X (p-*Hydroxybenzylpenilloaldehyde*, p-*hydroxyphenylacetamidoacetaldehyde*)

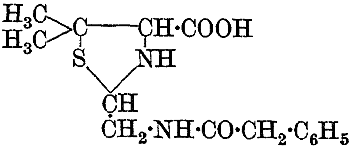


$C_{10}H_{11}O_3N$ MW, 193

Di-Et acetal: oil. Decomp. on dist. O-*Acetyl*: needles from Et_2O. M.p. 76°. *Benzyl ester*: cryst. from light petroleum. M.p. 75–6°.
2 : 4-*Dinitrophenylhydrazone*: needles from EtOH. M.p. 195–6°. O-*Acetyl*: needles from $MeOH–C_6H_6$. M.p. 207° decomp.

Brown, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 484.

Penilloic-G Acid (*Benzylpenilloic acid*)



$C_{15}H_{20}O_3N_2S$ MW, 308

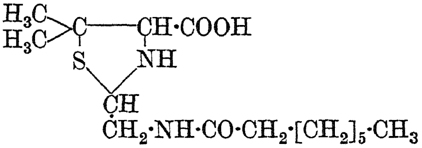
d-.
Two forms. (α) Needles + $1H_2O$ from H_2O. M.p. 112–14°, $[\alpha]_D$ + 57° in EtOH; anhyd., m.p. 87–92°, $[\alpha]_D^{23}$ + 53° in EtOH, + 109° in pH 8 buffer. (β) Anhyd. cryst. M.p. 158–60°. $[\alpha]_D^{23}$ + 26° in EtOH.
B,HCl: prisms from $MeOH–Et_2O$. M.p. 194–5° decomp. $[\alpha]_D^{18}$ − 70° in EtOH.

l-.
Needles from EtOH. M.p. 102–4° after sintering at 99°. $[\alpha]_D^{28}$ − 75° in MeOH. Very sol. Me_2CO, AcOEt, $CHCl_3$. Insol. CCl_4.
B,HCl: prisms from $MeOH–Et_2O$. M.p. 194–5° decomp. $[\alpha]_D^{18}$ − 70° in EtOH.

dl-.
Cryst. + $1H_2O$. M.p. 152–5° (90–9°).
B,HCl: prisms from $AcOH–Et_2O$. M.p. 180–2°. pK, 1·75 and 4·75 in H_2O at 23°.

Mozingo, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 535.

Penilloic-K Acid (*Heptylpenilloic acid*)

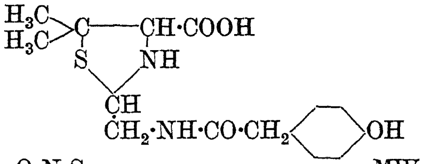


$C_{15}H_{28}O_3N_2S$ MW, 316

d-.
B,HCl: cryst. from $EtOH–Et_2O$. M.p. 190–1°. $[\alpha]_D^{25}$ + 81° in MeOH.

Cook, Heilbron, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 921.

Penilloic-X Acid (p-*Hydroxybenzylpenilloic acid*)

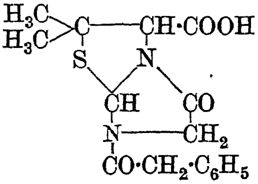


$C_{15}H_{20}O_4N_2S$ MW, 324

d-.
O-*Acetyl*: *B,HCl*, rosettes from $MeOH–Et_2O$. M.p. 198–9° decomp.
Carbobenzyloxy: needles + 1EtOH from EtOH. M.p. 106–8° decomp.

Mozingo, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 535.

Penillonic-G Acid (*Benzylpenillonic acid*)



$C_{16}H_{18}O_4N_2S$ MW, 334

Cryst. from Et_2O. M.p. 186–187·5°. $[\alpha]_D$ + 342° in MeOH. pK 3·0.
Na salt: white powder. $[\alpha]_D$ + 306° in H_2O.
Me ester: thermal inactivation product of penicillin Me ester. Cryst. from Et_2O. M.p. 152–4°. $[\alpha]_D$ + 131° in MeOH. Shows ultraviolet absorption max. at 257·5 mμ (ε 470).

Peck, Folkers, *The Chemistry of Penicillin*, Princeton University Press, 1949, p. 158.

Penta-acetylglucose.
See under Glucose.

Pentabromoacetone

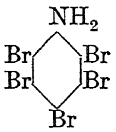
$CBr_3·CO·CHBr_2$

C_3HOBr_5 MW, 453

Needles from H_2O or EtOH, prisms from Et_2O. M.p. 79–80° (72°). Very sol. org. solvents. Insol. cold H_2O. Sublimes. Volatile in steam.

Jackson, Adams, *J. Am. Chem. Soc.*, 1915, **37**, 2533.

Pentabromoaniline



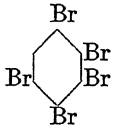
$C_6H_2NBr_5$ MW, 488

Needles from EtOH–toluene(1 : 2). M.p. 261–2°.

Jacobson, Löb, *Ber.*, 1900, **33**, 705.
Hantzsch, Smythe, *ibid.*, 520.

Pentabromoanisole.
See under Pentabromophenol.

Pentabromobenzene

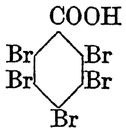


C_6HBr_5 MW, 473

Needles from AcOH or EtOH. M.p. 159–60°. Mod. sol. $CHCl_3$, C_6H_6. Spar. sol. EtOH, Et_2O, AcOH, ligroin. Very spar. volatile in steam.

Jacobson, Löb, *Ber.*, 1900, **33**, 702.
Eckert, Steiner, *Monatsh.*, 1915, **36**, 278.
Eckert, *J. prakt. Chem.*, 1921, **102**, 362.
I.G., D.R.P., 595,461, (*Chem. Zentr.*, 1934, II, 1030).

Pentabromobenzoic Acid (*Perbromobenzoic acid*)



$C_7HO_2Br_5$ MW, 517

Leaflets or needles from EtOH.Aq. M.p. 252° (234–5°). Sol. hot H_2O, EtOH. Spar. sol. pet. ether.

Nitrile: C_7NBr_5. MW, 498. Needles from C_6H_6–EtOH. Does not melt below 300°. Spar. sol. EtOH, Et_2O. Sublimes with slight decomp.

Merz, Weith, *Ber.*, 1883, **16**, 2892.
Blanksma, *Chem. Zentr.*, 1912, II, 1965.

Pentabromoethane

$CBr_3 \cdot CHBr_2$

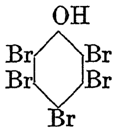
C_2HBr_5 MW, 425

Prisms. M.p. 56–7° (54°). B.p. 210°/300 mm. decomp. Very sol. Et_2O. Sol. EtOH.

Bourgoin, *Bull. soc. chim.*, 1875, **23**, 173.
Hunter, Edgar, *J. Am. Chem. Soc.*, 1932, **54**, 2025.

Pentabromophenetole.
See under Pentabromophenol.

Pentabromophenol



C_6HOBr_5 MW, 489

Needles from EtOH or CS_2. M.p. 229·5° (225°). Sublimes. pK = 8·0 in MeOH.

Me ether: pentabromoanisole. $C_7H_3OBr_5$. MW, 503. Needles from EtOH. M.p. 173–4°.

Et ether: pentabromophenetole. $C_8H_5OBr_5$. MW, 517. Needles from EtOH. M.p. 136°.

Propyl ether: $C_9H_7OBr_5$. MW, 531. Needles from EtOH. M.p. 98°.

Isopropyl ether: needles from ligroin. M.p. 86°.

n-*Butyl ether*: $C_{10}H_9OBr_5$. MW, 545. M.p. 79–80°.

sec.-n-*Butyl ether*: m.p. 57–8°.

Isobutyl ether: needles from ligroin. M.p. 92–3°.

Isoamyl ether: $C_{11}H_{11}OBr_5$. MW, 559. Needles from ligroin. M.p. 64–5°.

Allyl ether: $C_9H_5OBr_5$. MW, 529. Needles from ligroin. M.p. 167–8°.

Acetyl: prisms from EtOH. M.p. 197° (171°).

Zincke, Birschel, *Ann.*, 1908, **362**, 227.
Bonneaud, *Bull. soc. chim.*, 1910, **7**, 776.
Kohn, Fink, *Monatsh.*, 1923, **44**, 187.
Lucas, Kemp, *J. Am. Chem. Soc.*, 1921, **43**, 1654.
Raiford, Howland, *J. Am. Chem. Soc.*, 1931, **53**, 1054.

1 : 1 : 1 : 2 : 2-Pentabromopropane

$CH_3 \cdot CBr_2 \cdot CBr_3$

$C_3H_3Br_5$ MW, 439

Needles from C_6H_6. M.p. 212°.

Loevenich, Loser, Dierichs, *Ber.*, 1927, **60**, 955.

1 : 1 : 2 : 3 : 3-Pentabromopropane

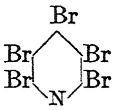
$CHBr_2 \cdot CHBr \cdot CHBr_2$

$C_3H_3Br_5$ MW, 439

Liq. B.p. 165–75°/17 mm. (163–5°/18 mm.).

Prins, D.R.P., 261,689, (*Chem. Abstracts*, 1913, II, 394).
Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 809.

Pentabromopyridine

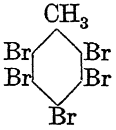


C_5NBr_5 MW, 474

Cryst. from C_6H_6. M.p. 209·5–210°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

Pentabromotoluene



$C_7H_3Br_5$ MW, 487

Needles from C_6H_6. M.p. 282 (280°). D^{17} 2·97. Spar. sol. EtOH, AcOH. Sol. about 102 parts C_6H_6 at 20°.

Bodroux, Taboury, *Bull. soc. chim.*, 1912, **11**, 395.
Datta, Chatterjee, *J. Am. Chem. Soc.*, 1916, **38**, 2548.
Bodroux, *Ann. chim.*, 1929, **11**, 547.

Pentacene.
See 2 : 3 : 6 : 7-Dibenzanthracene.

Pentachloroacetone

$CCl_3 \cdot CO \cdot CHCl_2$

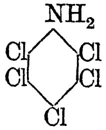
C_3HOCl_5 MW, 230·5

Liq. with odour resembling that of chloral. B.p. 192°/753 mm. D^{15} 1·69. Sol. 10 parts H_2O. Volatile in steam.

B,4H₂O: plates. M.p. 15–17°.

Fritsch, *Ber.*, 1893, **26**, 598; *Ann.*, 1894, **279**, 317.
Staedler, *Ann.*, 1859, **111**, 293.
Buc, U.S.P., 1,391,758, (*Chem. Zentr.*, 1922, IV, 940).

Pentachloroaniline



$C_6H_2NCl_5$ MW, 265·5

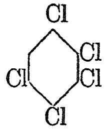
Needles from EtOH. M.p. 232°. Sol. EtOH, Et_2O. Mod. sol. ligroin.

N-*Acetyl*: N-*Me*, m.p. 136–7°. N-*Et*: m.p. 99–100°.

Willegerodt, Wilcke, *Ber.*, 1910, **43**, 2754.
Badische, D.R.P., 176,474, (*Chem. Zentr.*, 1907, I, 142).
Durand, Huguenin, B.P. 217,753, (*Chem. Zentr.*, 1925, I, 301).
Quist, Salo, *Chem. Zentr.*, 1934, II, 594.

Pentachloroanisole.
See under Pentachlorophenol.

Pentachlorobenzene

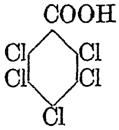


C_6HCl_5 MW, 250·5

Needles from EtOH. M.p. 86° (84°). B.p. 275–7°. D^{10} 1·8422, $D^{16\cdot5}$ 1·8342. Mod. sol. Et_2O, $CHCl_3$, C_6H_6, CS_2, CCl_4. Insol. cold EtOH.

Eckert, Steiner, *Ber.*, 1914, **47**, 2629.
v. der Linden, *Ber.*, 1912, **45**, 413.
Hollemann, *Rec. trav. chim.*, 1920, **39**, 736.

Pentachlorobenzoic Acid (*Perchlorobenzoic acid*)



$C_7HO_2Cl_5$ MW, 294·5

Needles. M.p. 201° (208°). Sublimes in vacuo with slight decomp.

Me ester: $C_8H_3O_2Cl_5$. MW, 308·5. Cryst. from MeOH. M.p. 97°.

Chloride: C_7OCl_6. MW, 313. Cryst. from EtOH. M.p. 87°.

Steiner, *Monatsh.*, 1915, **36**, 827.
Merz, Weith, *Ber.*, 1883, **16**, 2885.
Kirpal, Kunze, *Ber.*, 1929, **62**, 2102.

1 : 2 : 2 : 3 : 4-Pentachlorobutane

$CH_2Cl \cdot CHCl \cdot CCl_2 \cdot CH_2Cl$

$C_4H_5Cl_5$ MW, 2305.

Liq. B.p. 85°/10 mm. D_4^{20} 1·5543. n_D^{20} 1·5157.

du Pont, U.S.P., 1,964,720, (*Chem. Zentr.*, 1934, II, 3180).
Carothers, Berehet, *J. Am. Chem. Soc.*, 1933, **55**, 1628.

Pentachloroethane (*Pentalin*)

$CHCl_2 \cdot CCl_3$

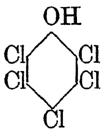
C_2HCl_5 MW, 202·5

Liq. with odour of chloroform. F.p. — 29° (— 22°). B.p. 162·00°, 152·2°/644 mm., 93·6°/103 mm., 69·0°/37 mm. D_4^{15} 1·6846, D_4^{25} 1·6712. n_D^{15} 1·50542. $AlCl_3 \longrightarrow$ tetrachloroethylene.

Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 260.
Salzbergwerk Neustassfurt, D.R.P., 248,982, (*Chem. Zentr.*, 1912, II, 299).
Timmermans, Martin, *J. chim. phys.*, 1926, **23**, 747.
Guyot, *Chimie et Industrie*, 1923, **10**, 13.

Pentachlorophenetole.
See under Pentachlorophenol.

Pentachlorophenol



C_6HOCl_5 MW, 266·5

Cryst. + H_2O from EtOH, needles from C_6H_6. M.p. 174°, anhyd. 191°. B.p. 309–10°/754·3 mm. D^{22} 1·978. Very sol. EtOH, Et_2O. Mod. sol. C_6H_6. Spar. sol. cold ligroin. Sublimes in long needles. pK = 8·2 in MeOH.

Me ether: pentachloroanisole. $C_7H_3OCl_5$. MW, 280·5. Needles from MeOH. M.p. 108°. B.p. 289°/745 mm. decomp. Sol. EtOH. Sublimes readily.

Et ether: pentachlorophenetole. $C_8H_5OCl_5$. MW, 294·5. Prisms from EtOH. M.p. 89–90°. Spar. sol. H_2O.

Propyl ether: $C_9H_7OCl_5$. MW, 308·5. Prisms from EtOH. M.p. 49–50°. Very sol. H_2O.

Butyl ether: $C_{10}H_9OCl_5$. MW, 322·5. Cryst. M.p. 15·5–16·5°. B.p. 343°.

Acetyl: needles from EtOH. M.p. 149·5–150·5° (147–8°). Sol. warm EtOH. Sublimes.

Propionyl: needles from ligroin. M.p. 78·5°. Very sol. Et_2O, C_6H_6.

Butyryl: needles. M.p. 59–62°.

Benzoyl: prisms. M.p. 164–5° (159–60°).

Barral, *Bull. soc. chim.*, 1895, **13**, 342.
Weber, Wolff, *Ber.*, 1885, **18**, 335.
Biltz, Giese, *Ber.*, 1904, **37**, 4019.
Barral, Jambon, *Bull. soc. chim.*, 1900, **23**, 823.
Mathieson, McCombie, *J. Chem. Soc.*, 1931, 1110.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.
Tomcsik, *J. pharm. chim.*, 1930, **11**, 101.
I.G., D.R.P., 527,393, (*Chem. Zentr.*, 1931, II, 2785).

1 : 1 : 1 : 2 : 3-Pentachloropropane

$CH_2Cl·CHCl·CCl_3$

$C_3H_3Cl_5$ MW, 216·5

Needles from hot EtOH. M.p. 179–80°. Sublimes.

Vitoria, *Rec. trav. chim.*, 1905, **24**, 282.
Henry, *ibid.*, 342.

1 : 1 : 2 : 2 : 3-Pentachloropropane

$CH_2Cl·CCl_2·CHCl_2$

$C_3H_3Cl_5$ MW, 216·5

Liq. with pleasant camphoraceous odour. B.p. 191–191·5°/755 mm. D_4^{25} 1·6326. n_D^{25} 1·5098.

Stevens, *J. Am. Chem. Soc.*, 1946, **68**, 622.

1 : 1 : 2 : 3 : 3-Pentachloropropane

$CHCl_2·CHCl·CHCl_2$

$C_3H_3Cl_5$ MW, 216·5

Liq. B.p. 198–200°, 126°/90 mm. D_4^{34} 1·6086. $n_D^{16·5}$ 1·5131.

Prins, *J. prakt. Chem.*, 1914, **89**, 421; D.R.P., 261,689, (*Chem. Zentr.*, 1913, II, 394).
Prins, Engelhard, *Rec. trav. chim.*, 1935, **54**, 307.

1 : 1 : 2 : 3 : 3-Pentachloropropylene

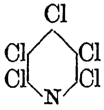
$CHCl_2·CCl:CCl_2$

C_3HCl_5 MW, 214·5

Liq. B.p. 183°, 116°/90 mm. D_4^{34} 1·6317. n_D^{20} 1·5313.

Prins, *Rec. trav. chim.*, 1946, **65**, 460.

Pentachloropyridine

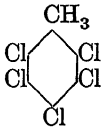


C_5NCl_5 MW, 251·5

Plates or needles from EtOH. M.p. 124°. Mod. sol. hot EtOH. Spar. sol. Et_2O. Insol. H_2O. Volatile in steam. Possesses no basic properties.

Sell, Dootson, *J. Chem. Soc.*, 1897, **71**, 1082; 1898, **73**, 441.
Hertog, Wibaut, Ley, *Rec. trav. chim.*, 1932, **51**, 381.

Pentachlorotoluene



$C_7H_3Cl_5$ MW, 264·5

Needles from C_6H_6. M.p. 218°. B.p. 301°. Sol. boiling C_6H_6. Spar. sol. CS_2. Very spar. sol. EtOH, Et_2O.

Beilstein, Kuhlberg, *Ann.*, 1869, **150**, 298.
Fichter, Glantzstein, *Ber.*, 1916, **49**, 2486.

Pentacosanal

$CH_3·[CH_2]_{23}·CHO$

$C_{25}H_{50}O$ MW, 366

M.p. 72–6°.

Semicarbazone: cryst. from AcOEt. M.p. 115–115·5°.

p-*Nitrophenylhydrazone*: cryst. from AcOH or MeOH. M.p. 104–5°.

Reindel *et al.*, *Ann.*, 1940, **544**, 116.

Pentacosane

$CH_3·[CH_2]_{23}·CH_3$

$C_{25}H_{52}$ MW, 352

Pearly leaflets from EtOH. M.p. 55·5–56°.

Brigl, *Z. physiol. Chem.*, 1915, **95**, 179.

The following pentacosanes have also been described :—

1. From paraffin oil. M.p. 53·8–54°. B.p. 152·5°/high vacuum.

Krafft, *Ber.*, 1907, **40**, 4783.

2. From paraffin oil. M.p. 54°. B.p. 282–4°/40 mm. D^{60} 0·7911, D^{90} 0·7854.

Mabery, *Am. Chem. J.*, 1905, **33**, 288.

3. From high-boiling tar. M.p. 53·5–54°. B.p. 254°/15 mm. n_D^{65} 1·43202, n_D^{80} 1·42624.

Gluud, *Ber.*, 1919, **52**, 1040, 1049.

Pentacosanic Acid

$CH_3{\cdot}[CH_2]_{22}{\cdot}CH_2{\cdot}COOH$

$C_{25}H_{50}O_2$ MW, 382

Cryst. from Me_2CO. M.p. 84–5°.

Me ester : $C_{26}H_{52}O_2$. MW, 396. Cryst. from Me_2CO. M.p. 61–2°.

Et ester : $C_{27}H_{54}O_2$. MW, 410. Cryst. from Me_2CO. M.p. 58–9°. B.p. 216–17°/0·5 mm.

Nitrile : *n*-tetracosyl cyanide. $C_{25}H_{49}N$. MW, 363. Cryst. from Me_2CO. M.p. 58–9°.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.

Pentacosanol-1.
See Pentacosyl Alcohol.

Pentacosanone-9.
See Octyl hexadecyl Ketone.

Pentacosyl Alcohol (*Pentacosanol*-1)

$CH_3{\cdot}[CH_2]_{23}{\cdot}CH_2OH$

$C_{25}H_{52}O$ MW, 368

Cryst. from Me_2CO. M.p. 78·5–79·5°. B.p. 214–16°/0·36 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 916.

Pentadecamethyleneimine

$CH_2{\cdot}[CH_2]_6{\cdot}CH_2$
$CH_2{\cdot}[CH_2]_5{\cdot}CH_2$ >NH

$C_{15}H_{29}N$ MW, 225

Cryst. M.p. 44–5°. pK 9·26.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1949, **32**, 544.

Pentadecanal.
See Pentadecylic Aldehyde.

Pentadecane

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH_3$

$C_{15}H_{32}$ MW, 212

Cryst. M.p. 10°. B.p. 270·5°/760 mm., 194°/100 mm., 160°/30 mm., 144°/15 mm. D_4^{20} 0·7689. n_D^{20} 1·43199.

Krafft, *Ber.*, 1882, **15**, 1700.
Mai, *Ber.*, 1889, **22**, 2134.
Vogel, *J. Chem. Soc.*, 1946, 137.

Pentadecane-1 : 1-dicarboxylic Acid.
See Tetradecylmalonic Acid.

Pentadecanoic Acid.
See Pentadecylic Acid.

Pentadecanoic Aldehyde.
See Pentadecylic Aldehyde.

Pentadecanol-1.
See Pentadecyl Alcohol.

Pentadecanol-3.
See Ethyldodecylcarbinol.

Pentadecanol-8.
See Di-*n*-heptylcarbinol.

Pentadecanone-2.
See Methyl tridecyl Ketone.

Pentadecanone-3.
See Ethyl dodecyl Ketone.

Pentadecene.
See Pentadecylene.

1-Pentadecenol-3 (3-*Hydroxy*-1-*pentadecylene, dodecylvinylcarbinol*)

$CH_3{\cdot}[CH_2]_{11}{\cdot}CH(OH){\cdot}CH{:}CH_2$

$C_{15}H_{30}O$ MW, 226

Cryst. M.p. 27–8°. B.p. 150–2°/6 mm. $D_4^{32\cdot5}$ 0·834. $n_D^{32\cdot5}$ 1·4481.

Delaby, Lecomte, *Bull. soc. chim.*, 1937, **4**, 742.

1-Pentadecenol-15 (*Pentadecylenic alcohol*, 14-*pentadecenyl alcohol*, 15-*hydroxy*-1-*pentadecylene*)

$HO{\cdot}CH_2{\cdot}[CH_2]_{12}{\cdot}CH{:}CH_2$

$C_{15}H_{30}O$ MW, 226

Leaflets from pet. ether. M.p. 32–3°. B.p. 170–2°/10 mm.

Phenylurethane : leaflets from EtOH. M.p. 60·5–61°.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 128.

2-Pentadecenol-15 (13-*Pentadecenol*-1, *isopentadecylenic alcohol*, 13-*pentadecenyl alcohol*, 15-*hydroxy*-2-*pentadecylene*)

$HO{\cdot}CH_2{\cdot}[CH_2]_{11}{\cdot}CH{:}CH{\cdot}CH_3$

$C_{15}H_{30}O$ MW, 226

Leaflets from pet. ether. M.p. 40–40·5°. B.p. 170–2°/8 mm.

Phenylurethane : leaflets from EtOH. M.p. 68–68·4°.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 130.

Pentadecenyl Alcohol.
See Pentadecenol.

Pentadecenylsalicylic Acid.
See Ginkgolic Acid.

Pentadecyl Alcohol (*Pentadecanol*-1)

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH_2OH$

$C_{15}H_{32}O$ MW, 228

Cryst. M.p. 45–6° (44°).

Formyl : cryst. M.p. 13·69°. B.p. 201·5°/30 mm. D_4^{25} 0·8618. n_D^{20} 1·4399.

Palmityl : cryst. from EtOH. M.p. 55·5°.
p-*Nitrobenzoyl* : cryst. from pet. ether. M.p. 45·8°.
Phenylurethane : plates from C_6H_6. M.p. 72°.
α-*Naphthylurethane* : needles from pet. ether. M.p. 85°.

Simonini, *Monatsh.*, 1893, **14**, 85.
Gascard, *Ann. chim.*, 1921, **15**, 332.
Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, **55**, 3825.
Dauben, *J. Am. Chem. Soc.*, 1948, **70**, 1377.

Pentadecylamine (1-*Amino*-n-*pentadecane*)

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH_2NH_2$

$C_{15}H_{33}N$ MW, 227

Leaflets or needles. M.p. 36·5° (33°). B.p. 298–301° (301°/720 mm.), 107–10° (120·5°)/1 mm. Sol. EtOH, Et_2O. $k = 4{\cdot}1 \times 10^{-4}$ at 25°.
B,HCl : plates from EtOH. M.p. 199°.
Acetyl : needles from pet. ether. M.p. 72°. Sol. EtOH, Et_2O, hot ligroin. Insol. H_2O.

Naegeli, Grüntuch, Lendorff, *Helv. Chim. Acta*, 1929, **12**, 240.

3-Pentadecylcatechol.
See Hydrourushiol.

1-Pentadecylene (1-*Pentadecene*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH{:}CH_2$

$C_{15}H_{30}$ MW, 210

M.p. − 2·8°. B.p. 127·5–128·5°/10 mm. D_4^0 0·7921, D_4^{25} 0·7751. n_D^{25} 1·4353.

Kozacik, Reid, *J. Am. Chem. Soc.*, 1938, **60**, 2436.
Schmidt, Schoeller, Eberlein, *Ber.*, 1941, **74**, 1313.

7-Pentadecylene (7-*Pentadecene*)

$CH_3{\cdot}[CH_2]_6{\cdot}CH{:}CH{\cdot}[CH_2]_5{\cdot}CH_3$

$C_{15}H_{30}$ MW, 210

B.p. 114°/3·2 mm. D^{20} 0·7765. n_D^{20} 1·4420.

Asinger, Eckoldt, *Ber.*, 1943, **76**, 585.

Pentadecylene-carboxylic Acid.
See Gaidic Acid *and* Palmitoleic Acid.
Pentadecylene-dicarboxylic Acid.
See Civetic Acid.
Pentadecylenic Alcohol.
See Pentadecenol.
Pentadecyl *p*-hydroxyphenyl Ketone.
See *p*-Hydroxypalmitophenone.

Pentadecylic Acid (*Pentadecanoic acid, tetradecane*-1-*carboxylic acid*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2{\cdot}COOH$

$C_{15}H_{30}O_2$ MW, 242

Plates from Me_2CO.Aq. M.p. 53–4° (51°). B.p. 257°/100 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, pet. ether.
Me ester : $C_{16}H_{32}O_2$. MW, 256. Needles from EtOH.Aq. M.p. 18·5°.
Et ester : $C_{17}H_{34}O_2$. MW, 270. Needles from EtOH.Aq. M.p. 14°. Sol. most org. solvents.
p-*Nitrobenzyl ester* : m.p. 48·5–49°.
p-*Phenylphenacyl ester* : m.p. 91·3–91·8°.
Chloride : $C_{15}H_{29}OCl$. MW, 260·5. Liq. B.p. 157°/5 mm.
Amide : $C_{15}H_{31}ON$. MW, 241. Needles from EtOH. M.p. 102·5°. Sol. boiling EtOH. Mod. sol. Et_2O. Insol. cold H_2O, pet. ether.
p-*Toluidide* : m.p. 92–3°.

Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1898.
Majima, Nakamura, *Ber.*, 1913, **46**, 4094.
Ford-Moore, Phillips, *Rec. trav. chim.*, 1934, **53**, 857.

Pentadecylic Aldehyde (*Pentadecanal, pentadecanoic aldehyde*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2{\cdot}CHO$

$C_{15}H_{30}O$ MW, 226

Needles. M.p. 24–5°. B.p. 185°/25 mm., 172–6°/15 mm. Polymerises readily.
Polymer : m.p. 69–70°.
Oxime : needles from EtOH.Aq. M.p. 86°. Sol. Et_2O. Spar. sol. EtOH, C_6H_6, pet. ether.
Semicarbazone : m.p. 108–9°.
Thiosemicarbazone : cryst. from Et_2O. M.p. 95–96·5°. Spar. sol. EtOH, Me_2CO, AcOEt, C_6H_6. Insol. H_2O, pet. ether.
p-*Bromophenylhydrazone* : cryst. M.p. 40–50°. Unstable.
p-*Nitrophenylhydrazone* : yellow plates from EtOH. M.p. 94–5°. Sol. EtOH, Me_2CO, AcOH, AcOEt, CCl_4, C_6H_6.
2 : 4-*Dinitrophenylhydrazone* : m.p. 107·5°. Sol. AcOEt, $CHCl_3$, C_6H_6, hot Et_2O, Me_2CO, AcOH, CCl_4.
Benzoylhydrazone : plates from EtOH. M.p. 87°.
m-*Nitrobenzoylhydrazone* : plates from EtOH. M.p. 102°. Spar. sol. Et_2O.
Cyanhydrin : *see under* 1-Hydroxypalmitic Acid.

Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1896.
Landa, *Bull soc. chim.*, 1925, **37**, 1235.
Lauer, Gensler, Miller, *J. Am. Chem. Soc.*, 1941, **63**, 1154.

Pentadecyl 2-naphthyl Ketone.
See 2-Palmitonaphthone.
3-Pentadecylphenol.
See Hydroginkgol.
Pentadecyl phenyl Ketone.
See Palmitophenone.
5-Pentadecylresorcinol.
See Hydrobilobol.
6-Pentadecylsalicylic Acid.
See Hydroginkgolic Acid.

1 : 2-Pentadiene (*Ethylallene, propylidene-ethylene*)

$CH_3{\cdot}CH_2{\cdot}CH{:}C{:}CH_2$

C_5H_8 MW, 68

Mobile liq. B.p. 44–5°. D^{20} 0·6890. n_D^{20} 1·4149.

Bouis, *Bull. soc. chim.*, 1927, **41**, 1160.

1 : 3-Pentadiene (1-*Methylbutadiene*-1 : 3-, 1-*methyldivinyl*, 1-*methylerythrene*, *piperylene*)

$CH_3{\cdot}CH{:}CH{\cdot}CH{:}CH_2$

C_5H_8 MW, 68

Liq. B.p. 42°. $D_4^{13\cdot6}$ 0·6887, D_4^{20} 0·6830. $n_D^{13\cdot6}$ 1·43443, n_D^{20} 1·4280.

Auwers, Westermann, *Ber.*, 1921, **54**, 2993.
Prévost, *Ann. chim.*, 1928, **10**, 173.
Farmer, Warren, *J. Chem. Soc.*, 1931, 3221.
Schniepp, Geller, *J. Am. Chem. Soc.*, 1945, **67**, 55.

Cis-isomer: b.p. 43·5°. n_D^{20} 1·4360.
Trans-isomer: b.p. 42°. n_D^{20} 1·4300.

Frank, Emmick, Johnson, *J. Am. Chem. Soc.*, 1947, **69**, 2313.

1 : 4-Pentadiene (*Divinylmethane*, *allylethylene*)

$CH_2{:}CH{\cdot}CH_2{\cdot}CH{:}CH_2$

C_5H_8 MW, 68

Liq. B.p. 25·8–26·2°/756 mm. D_4^{20} 0·6594. n_D^{20} 1·3883.

Kogerman, *J. Am. Chem. Soc.*, 1930, **52**, 5060.
Shoemaker, Boord, *J. Am. Chem. Soc.*, 1931, **53**, 1505.
Schniepp, Geller, *J. Am. Chem. Soc.*, 1945, **67**, 55.

2 : 3-Pentadiene.
See sym.-Dimethylallene.

1 : 3-Pentadiene-1-carboxylic Acid.
See Sorbic Acid.

1 : 3-Pentadienol-5 (5-*Hydroxy*-1 : 3-*pentadiene*)

$HO{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH{:}CH_2$

C_5H_8O MW, 84

B.p. 95–7°/100 mm. n_D^{18} 1·4902.
α-*Naphthylurethane*: needles. M.p. 97·5°.

Heilbron, Jones, McCombie, Weedon, *J. Chem. Soc.*, 1945, 84.
Heilbron, Jones, Sondheimer, *J. Chem. Soc.*, 1947, 1586.

1 : 4-Pentadienol-3 (3-*Hydroxy*-1 : 4-*pentadiene*)

$CH_2{:}CH{\cdot}CH(OH){\cdot}CH{:}CH_2$

C_5H_8O MW, 84

B.p. 64–6°/100 mm. n_D^{17} 1·4400.
α-*Naphthylurethane*: needles from MeOH.Aq. M.p. 100–1°.

Heilbron, Jones, McCombie, Weedon, *J. Chem. Soc.*, 1945, 84.

1 : 3-Pentadi-yne (*Methyldiacetylene*)

$CH_3{\cdot}C{:}C{\cdot}C{:}CH$

C_5H_4 MW, 64

B.p. 54–6°. D_4^{21} 0·7375. n_D^{21} 1·4431.

Prévost, *Ann. chim.*, 1928, **10**, 356.

Pentaerythritol (*Tetrahydroxymethylmethane*, *tetramethylolmethane*, *tetrahydroxytetramethylmethane*)

$$\begin{matrix}HO{\cdot}CH_2\\HO{\cdot}CH_2\end{matrix}\!>C<\!\begin{matrix}CH_2OH\\CH_2OH\end{matrix}$$

$C_5H_{12}O_4$ MW, 136

Cryst. from dil. HCl. M.p. 260° (253°). Sol. 18 parts H_2O at 15°. Heat of comb. C_p 661·4 Cal. $HNO_3 \longrightarrow$ oxalic acid. $CrO_3 \longrightarrow$ $H{\cdot}COOH + CO_2$.
Tetra-Et ether: $C_{13}H_{28}O_4$. MW, 248. Oil. B.p. 220–5°. D_4^0 0·9229, D_4^{16} 0·9082.
Tetra-acetyl: needles from H_2O or C_6H_6. M.p. 82–3°.
Tetranitrate: niperyt, penthrit. Prisms from Me_2CO–EtOH. M.p. 138–40°. Sol. Me_2CO. Spar. sol. EtOH, Et_2O. Reduces Fehling's.

Schwink, *Organic Syntheses*, Collective Vol. I, 417.
Stellbacher, *Chem. Zentr.*, 1919, II, 261.
Deutsche Gold- und Silber-Scheideanstallt vorm. Roessler, D.R.P., 596,509, (*Chem. Zentr.*, 1933, II, 2190).
Friederich, Brün, *Ber.*, 1930, **63**, 2681.
Bincer, Hess, *Ber.*, 1928, **61**, 539.
Clarke, U.S.P., 1,583,658, (*Chem. Zentr.*, 1926, II, 1191).
Desvergnes, *Chem. Abstracts*, 1933, **27**, 4675.
Dumontel, Giovetti, *Industria chimica*, 1934, **9**, 767 (*Review*).

Pentaerythrityl tetrabromide (sym.-*Tetrabromoneopentane*, sym.-*tetrabromotetramethylmethane*)

$$\begin{matrix}BrCH_2\\BrCH_2\end{matrix}\!>C<\!\begin{matrix}CH_2Br\\CH_2Br\end{matrix}$$

$C_5H_8Br_4$ MW, 388

M.p. 163°.

Wagner, Dengel, *Z. physik. Chem.*, 1932, **16**, 384.

Pentaerythrityl tetrachloride (sym.-*Tetrachloroneopentane*, sym.-*tetrachlorotetramethylmethane*)

$$\begin{matrix}ClCH_2\\ClCH_2\end{matrix}\!>C<\!\begin{matrix}CH_2Cl\\CH_2Cl\end{matrix}$$

$C_5H_8Cl_4$ MW, 210

M.p. 97°. B.p. 110°/12 mm.

Wagner, Dengel, *Z. physik. Chem.*, 1932, **16**, 384.

Pentaerythrityl tetraiodide (sym.-*Tetraiodoneopentane*, sym.-*tetraiodotetramethylmethane*)

$$\begin{matrix}ICH_2\\ICH_2\end{matrix}\!>C<\!\begin{matrix}CH_2I\\CH_2I\end{matrix}$$

$C_5H_8I_4$ MW, 576

M.p. 225°.

Wagner, Dengel, *Z. physik. Chem.*, 1932, **16**, 384.

Pentafluoroiodoethane

C_2F_5I MW, 246

B.p. 13°.

Banks *et al.*, *J. Chem. Soc.*, 1948, 2188.

Pentahomocholine (5-*Dimethylamino*-n-*amyl alcohol*)

$(CH_3)_2N \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2OH$

$C_7H_{17}ON$ MW, 131

Liq. B.p. 115–16°/25 mm. Misc. with H_2O. Volatile in steam. Weak base.

Methochloride: cryst.

Methiodide: leaflets. M.p. 134°.

Methochloroplatinate: pale yellow needles from EtOH.Aq. M.p. 213°. Very sol. H_2O. Spar. sol. EtOH.

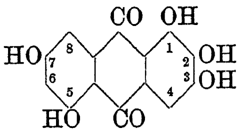
Methochloroaurate: brownish-yellow leaflets from H_2O. M.p. 147°.

v. Braun, *Ber.*, 1916, **49**, 976.

Pentahydroxy-1-aminohexane.

See Glucamine.

1 : 2 : 3 : 5 : 7-Pentahydroxyanthraquinone (5 : 7-*Dihydroxyanthragallol*)



$C_{14}H_8O_7$ MW, 288

Red cryst. from EtOH. Does not melt below 360°. Sol. boiling EtOH, Me_2CO. Spar. sol. Et_2O, AcOH. Insol. H_2O, $CHCl_3$, C_6H_6, ligroin.

Penta-acetyl: pale yellow needles from EtOH. M.p. 229°. Sol. hot EtOH, AcOH.

Noah, *Ber.*, 1886, **19**, 751.

Liebermann, v. Kostanecki, Noah, *Ann.*, 1887, **240**, 273.

1 : 2 : 4 : 5 : 8-Pentahydroxyanthraquinone (*Alizarin Cyanine R*)

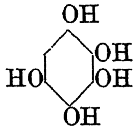
Bronze leaflets from $PhNO_2$. Distils undecomp. Conc. H_2SO_4 ⟶ blue sol. with red flour., + boric acid ⟶ greenish blue.

Penta-acetyl: leaflets from C_6H_6.

Bayer, D.R.P., 66,153.

Gattermann, *J. prakt. Chem.*, 1891, **43**, 250.

Pentahydroxybenzene



$C_6H_6O_5$ MW, 158

Two compounds of this formula are described in the literature.

1. Cryst. from conc. HI. Very sol. H_2O. Insol. org. solvents.

Wenzel, Weidel, *Chem. Zentr.*, 1903, II, 829.

2. Pale violet needles from AcOEt–C_6H_6. Blackens on heating. Sol. EtOH, Et_2O, AcOEt. Spar. sol. H_2O. Insol. C_6H_6. Alkalis ⟶ green sols. $FeCl_3$ ⟶ reddish-brown col.

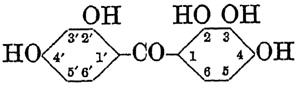
Penta-Me ether: nacreous rhombic plates from pet. ether. M.p. 58–9°. B.p. 150°/14 mm.

Penta-acetyl: needles from EtOH–pet. ether. M.p. 165° decomp. Sol. Et_2O. Spar. sol. H_2O, EtOH, pet. ether.

Einhorn, Cobliner, Pfeiffer, *Ber.*, 1904, **37**, 122.

Baker, *J. Chem. Soc.*, 1941, 662.

2 : 3 : 4 : 2′ : 4′-Pentahydroxybenzophenone



$C_{13}H_{10}O_6$ MW, 262

Yellowish needles + $2H_2O$ from EtOH. M.p. 187° (168–70°). Sol. H_2O ⟶ yellow col. ⟶ greenish-yellow ⟶ olive-green on standing.

Badische, D.R.P.s, 49,149, 50,451.

Atkinson, Heilbron, *J. Chem. Soc.*, 1926, 2690.

2 : 3 : 4 : 3′ : 4′-Pentahydroxybenzophenone.

Yellow needles + $2H_2O$ from boiling H_2O. M.p. anhyd. 192–3°. Sol. hot H_2O, EtOH, Me_2CO, AcOH. Spar. sol. Et_2O. Insol. C_6H_6. Conc. H_2SO_4 ⟶ orange sol.

Noelting, Meyer, *Ber.*, 1897, **30**, 2591.

2 : 3 : 4 : 3′ : 5′-Pentahydroxybenzophenone.

3 : 4 : 3′ : 5′-*Tetra-Me ether*: $C_{17}H_{18}O_6$. MW, 318. Pale yellow cryst. from EtOH. M.p. 123–4°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Spar. sol. AcOH, ligroin. Conc. H_2SO_4 ⟶ yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

2 : 4 : 6 : 2′ : 4′-Pentahydroxybenzophenone.

Penta-Me ether: $C_{18}H_{20}O_6$. MW, 332. Pale yellow leaflets from EtOH.Aq. M.p. 138°.

Tambor, *Ber.*, 1910, **43**, 1888.

2 : 4 : 6 : 2′ : 6′-Pentahydroxybenzophenone.

2′ : 6′-*Di-Me ether*: $C_{15}H_{14}O_6$. MW, 290. Plates from EtOH.Aq. M.p. 216–18°. Sol.

$EtOH$, $CHCl_3$, C_6H_6. Spar. sol. Et_2O. $FeCl_3$ ⟶ intense blue col.

Korczynski, Nowakowski, *Bull. soc. chim.*, 1928, **43**, 336.

2 : 4 : 6 : 3′ : 4′-Pentahydroxybenzophenone.
See Maclurin.

2 : 4 : 6 : 3′ : 5′-Pentahydroxybenzophenone.
Penta-Me ether : needles. M.p. 132–3°. Sol. Et_2O, AcOH, $CHCl_3$, C_6H_6, warm EtOH. Spar. sol. ligroin.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 409.

3 : 4 : 5 : 2′ : 4′-Pentahydroxybenzophenone.
Yellow needles from EtOH.Aq. M.p. 242°. Sol. most org. solvents. Spar. sol. H_2O. Alkalis ⟶ intense red col. Alc. $FeCl_3$ ⟶ green col.
3 : 4 : 5-*Tri-Me ether* : $C_{16}H_{16}O_6$. MW, 304. Yellow needles from EtOH.Aq. or H_2O. M.p. 165°. Conc. H_2SO_4 ⟶ intense yellow sol.

Bargellini, Grippa, *Gazz. chim. ital.*, 1927, **57**, 138.
Korczynski, Nowakowski, *Bull. soc. chim.*, 1928, **43**, 335.

3 : 4 : 5 : 3′ : 4′-Pentahydroxybenzophenone.
Yellow needles + $2H_2O$ from H_2O. M.p. anhyd. 266°. Sol. hot H_2O, EtOH, Me_2CO. Mod. sol. hot AcOH. Insol. Et_2O, C_6H_6. Conc. H_2SO_4 ⟶ red col. ⟶ protocatechuic acid on heating.
Penta-Me ether : needles from EtOH. M.p. 119–20°. Conc. H_2SO_4 ⟶ yellow sol. *Oxime* : needles from C_6H_6. M.p. 143°.

Noelting, Meyer, *Ber.*, 1897, **30**, 2591.
v. Kostanecki, Tambor, *Ber.*, 1906, **39**, 4026.
Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1664.

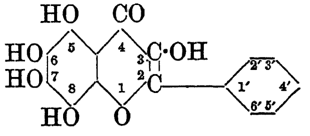
Pentahydroxycyclohexane.
See Quercitol.

Pentahydroxycyclohexanone.
See Inosose.

3 : 5 : 6 : 7 : 8 - Pentahydroxy - 2 - ethyl - 1 : 4-naphthoquinone.
See Echinochrome A.

3 : 5 : 7 : 3′ : 4′-Pentahydroxyflavan.
See Catechin.

3 : 5 : 6 : 7 : 8-Pentahydroxyflavone



$C_{15}H_{10}O_7$ MW, 302

Yellow narrow rectangular plates from AcOEt. M.p. 257–8°.
Penta-Me ether : long needles from EtOH.Aq. M.p. 81–2°.
Penta-acetyl : colourless flat needles from AcOEt. M.p. 207–8°.

Murti, Row, Seshadri, *Chem. Abstracts*, 1947, **41**, 2417.

3 : 5 : 6 : 7 : 4′-Pentahydroxyflavone.
Yellow micro-needles from AcOH.Aq. Darkens at 270°. Partially melts at 314–20° with decomp. Mg + acid alc. sol. ⟶ pinkish-red col. Alc. $Pb(OAc)_2$ ⟶ orange ppt. changing to greenish-brown. Alc. $FeCl_3$ ⟶ olive-green col.
3 : 6 : 7 : 4′-*Tetra-Me ether* : $C_{19}H_{18}O_7$. MW, 358. Yellow plates from EtOH. M.p. 171°. Alc. $FeCl_3$ ⟶ olive-green col.
Penta-Me ether : *see* Tangeritin.

Goldsworthy, Robinson, *J. Chem. Soc.*, 1937, 48.

3 : 5 : 7 : 8 : 4′-Pentahydroxyflavone.
See Herbacetin.

3 : 5 : 7 : 2′ : 4′-Pentahydroxyflavone.
See Morin.

3 : 5 : 7 : 3′ : 4′-Pentahydroxyflavone.
See Quercetin.

3 : 6 : 7 : 8 : 4′-Pentahydroxyflavone.
Small yellowish cryst. from EtOH. Begins to decomp. at 270–80° and not molten at 360°. Conc. H_2SO_4 ⟶ greenish-yellow col.
6 : 7 : 8 : 4′-*Tetra-Me ether* : pale yellow cryst. from EtOH. M.p. 187°. Conc. H_2SO_4 ⟶ golden-yellow col.
Penta-Me ether : small colourless needles from EtOH. M.p. 141°. Conc. H_2SO_4 ⟶ lemon-yellow col.
Penta-acetyl : small felted white needles from AcOH. M.p. 216°.

Bargellini, Oliverio, *Ber.*, 1942, **75**, 2088.

3 : 6 : 7 : 3′ : 4′-Pentahydroxyflavone.
Darkens at 315–20° and not molten below 310°. Bright green fluor. in EtOH. Conc. H_2SO_4 ⟶ bright yellow col. $FeCl_3$ in EtOH ⟶ dark green col.
6 : 7 : 3′ : 4′-*Tetra-Me ether* : cryst. from EtOH. M.p. 228°.
3 : 7 : 3′ : 4′-*Tetra-Me ether* : short yellow needles and plates from AcOH.Aq. M.p. 219–20°.
Penta-Me ether : fine crystals. M.p. 186–7°. Bluish-violet fluor. in EtOH. No col. with $FeCl_3$.
Penta-acetyl : m.p. 223–4°.

Row, Seshadri, *Chem. Abstracts*, 1945, **39**, 4325.

3 : 7 : 8 : 3′ : 4′-Pentahydroxyflavone.
Pale yellow needles + H_2O from EtOH.Aq.

M.p. 308° decomp. Conc. H_2SO_4 ⟶ intense yellow col. Dil. NaOH ⟶ reddish-yellow col.

7 : 8 : 3′ : 4′-*Tetra-Me ether* : $C_{19}H_{18}O_7$. MW, 358. Pale yellow needles from EtOH or AcOH–EtOH. M.p. 217°. Sol. hot AcOH. Spar. sol. EtOH. Conc. H_2SO_4 ⟶ intense yellow col. *Acetyl* : needles from EtOH.Aq. M.p. 176°.

Penta-acetyl : needles from EtOH. M.p. 172–3°.

v. Kostanecki, Rudse, *Ber.*, 1905, **38**, 938.

3 : 7 : 3′ : 4′ : 5′-Pentahydroxyflavone.

See Robinetin.

5 : 6 : 7 : 8 : 4′-Pentahydroxyflavone.

M.p. 318° decomp.

6 : 7 : 4′-*Tri-Me ether* : m.p. 250–1° decomp.

Penta-Me ether : m.p. 150–1°.

Murti, Rao, Seshadri, *Chem. Abstracts*, 1948, **42**, 5909.

5 : 7 : 3′ : 4′ : 5′-Pentahydroxyflavone.

See Tricetin.

7 : 8 : 3′ : 4′ : 5′-Pentahydroxyflavone.

Yellow needles from EtOH.Aq. Does not melt below 345°.

Penta-acetyl : needles from EtOH. M.p. 263°.

Badhwar, Kang, Venkataraman, *J. Chem. Soc.*, 1932, 1110.

Pentahydroxyhexylamine.

See Glucamine.

2 : 5 : 6 : 7 : 8-Pentahydroxy-3-(1-hydroxyethyl)-1 : 4-naphthoquinone.

See Spinochrome A.

Pentaketocyclopentane.

See Leuconic Acid.

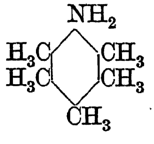
Pental.

See 2-Methyl-2-butylene.

Pentalin.

See Pentachloroethane.

Pentamethylaniline (*Aminopentamethylbenzene*)



$C_{11}H_{17}N$ MW, 163

Prismatic cryst. from EtOH. M.p. 151–2°. B.p. 277–8°. Sol. EtOH, Et_2O. Insol. H_2O.

N-*Me* : $C_{12}H_{19}N$. MW, 177. Cryst. from EtOH. M.p. 60–1°.

N-*Di-Me* : $C_{13}H_{21}N$. MW, 191. Cryst. M.p. 53–4°.

N-*Formyl* : needles from EtOH.Aq. M.p. 217°.

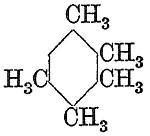
N-*Acetyl* : needles from EtOH. M.p. 213°.

Hoffmann, *Ber.*, 1885, **18**, 1822.
Limpach, *Ber.*, 1888, **21**, 645.
Willstätter, Kubli, *Ber.*, 1909, **42**, 4162.
Dimroth, Leichtlin, Friedemann, *Ber.*, 1917, **50**, 1543.

Pentamethylanisole.

See under Pentamethylphenol.

Pentamethylbenzene



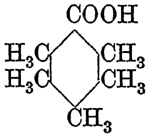
$C_{11}H_{16}$ MW, 148

Prisms from EtOH.Aq. or EtOH–C_6H_6. M.p. 53° (51·5°). B.p. 231°. Very sol. EtOH. Heat of comb. C_v 1551·8 Cal.

Picrate : golden-yellow prisms from EtOH. M.p. 131°.

Jacobsen, *Ber.*, 1887, **20**, 896.
v. Braun, Nelles, *Ber.*, 1934, **67**, 1099.
Smith, *Organic Syntheses*, 1930, X, 34.

Pentamethylbenzoic Acid



$C_{12}H_{16}O_2$ MW, 192

Needles from boiling H_2O; leaflets or needles from EtOH.Aq. M.p. 210·5°. Very sol. hot EtOH. Insol. cold H_2O. Volatile in steam. Sublimes. Fuming HNO_3 at 200° ⟶ pentamethylbenzene + CO_2.

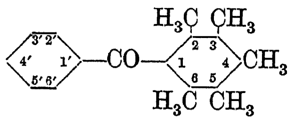
Me ester : $C_{13}H_{18}O_2$. MW, 206. Plates from MeOH. M.p. 67·5°. B.p. 299–300°.

Amide : $C_{12}H_{17}ON$. MW, 191. Leaflets from EtOH. M.p. 206°. Insol. H_2O.

Nitrile : $C_{12}H_{15}N$. MW, 173. Needles from EtOH. M.p. 170° (168°). B.p. 294–5° (290–2°). Very sol. hot EtOH. Insol. H_2O.

Jacobsen, *Ber.*, 1889, **22**, 1221.
Hoffmann, *Ber.*, 1885, **18**, 1825.
Clement, *Compt. rend.*, 1934, **198**, 666.

2 : 3 : 4 : 5 : 6-Pentamethylbenzophenone



$C_{18}H_{20}O$ MW, 252

Long needles. M.p. 125° (135–6°).

Semicarbazone : m.p. 170°.

Clement, *Ann. chim.*, 1940, **13**, 243.
Fuson, McKusick, Mills, *J. Org. Chem.*, 1946, **11**, 65.

2 : 4 : 5 : 3′ : 4′-Pentamethylbenzophenone.

Prisms from C_6H_6. M.p. 90°. B.p. 189–90°/3 mm. Very sol. most solvents.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2327.

2 : 4 : 6 : 2′ : 5′-Pentamethylbenzophenone.
White needles from EtOH. M.p. 77–8°.

Newman, Lord, *J. Am. Chem. Soc.*, 1944, **66**, 734.

2 : 4 : 6 : 2′ : 6′-Pentamethylbenzophenone.
M.p. 86°.

Coops *et al.*, *Rec. trav. chim.*, 1940, **59**, 1113.

2 : 4 : 6 : 3′ : 5′-Pentamethylbenzophenone.
Prisms from pet. ether. M.p. 84–5°. Sol. hot EtOH. Conc. $H_2SO_4 \longrightarrow$ yellow sol.

Weiler, *Ber.*, 1899, **32**, 1910; 1900, **33**, 344.

Pentamethylene.
See Cyclopentane.

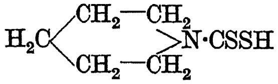
Pentamethylene-aniline.
See *N*-Phenylpiperidine.

Pentamethylene bromide.
See 1 : 5-Dibromo-*n*-pentane.

Pentamethylene chloride.
See 1 : 5-Dichloro-*n*-pentane.

Pentamethylenediamine.
See Cadaverine.

Pentamethylenedithiocarbamic Acid (*Piperidine* N-*dithiocarboxylic acid*)



$C_6H_{11}NS_2$ MW, 161

Free acid not known. Salts are powerful vulcanisation accelerators.

Piperidine salt: leaflets from EtOH. M.p. 174°. Well known rapid vulcanisation accelerator.

Phenylhydrazine salt: m.p. 218°.

Me ester: $C_7H_{13}NS_2$. MW, 175. Plates. M.p. 33–4°. B.p. about 260°. Very sol.

Phenyl ester: $C_{12}H_{15}NS_2$. MW, 237. M.p. 116–17.°

p-*Tolyl ester*: $C_{13}H_{17}NS_2$. MW, 251. M.p. 118–19°.

Losanitsch, *Ber.*, 1907, **40**, 2974.
Delépine, *Bull. soc. chim.*, 1902, **27**, 592.
Maas, Wolffenstein, *Ber.*, 1898, **31**, 2689.
Clifford, Lichty, *J. Am. Chem. Soc.*, 1932, **54**, 1166.
I.C.I., B.P. 358,230, (*Brit. Chem. Abstracts*, 1932, 138).

Pentamethyleneglycine.
See Piperidinoacetic Acid.

Pentamethylene Glycol (1 : 5-*Dihydroxypentane, pentandiol*-1 : 5)

$$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2OH$$

$C_5H_{12}O_2$ MW, 104

Oily liq. with bitter taste. M.p. — 18°. B.p. 238–9° (260°) 155°/31 mm., 134°/12 mm., 119–20°/3 mm. Sol. H_2O, EtOH. Spar. sol. Et_2O. D^0 1·004, D^{11}_{15} 0·999, D^{20}_{20} 0·9939. n^{20}_D 1·4499.

Di-Me ether: $C_7H_{16}O_2$. MW, 132. B.p. 156–7°/760 mm. (159°). D^{15} 0·8616. n^{15}_D 1·4094.

Di-isoamyl ether: $C_{15}H_{32}O_2$. MW, 244. Liq. B.p. 276–7°/759 mm., 159–60°/20 mm. D^{18} 0·844.

Phenyl ether: $C_{11}H_{16}O_2$. MW, 180. B.p. 150–5°/11 mm. *Phenylurethane*: m.p. 93°.

Diacetyl: liq. with fruity odour. F.p. 2°. B.p. 241°/760 mm., 122–3°/3 mm. D^{15} 1·0328, D^{18} 1·021. n^{15}_D 1·4282, n^{19}_D 1·4261.

Succinyl: m.p. 19°. B.p. 88–9°/1 mm. D^{60}_4 1·1373. n^{60}_D 1·4583. *Dimeric form*: m.p. 87°.

Di-p-*nitrobenzoyl*: cryst. from C_6H_6. M.p. 104·5°.

Carbonate: powder. M.p. 44–6°. Sol. Me_2CO, $CHCl_3$, AcOH, C_6H_6. Insol. EtOH, Et_2O, pet. ether. *Dimeric form*: m.p. 117–18°.

Di-phenylurethane: needles from EtOH or EtOH–$CHCl_3$. M.p. 176°.

Di-1-*naphthylurethane*: plates. M.p. 147°.

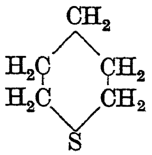
du Pont, U.S.P., 1,995,291, (*Chem. Abstracts*, 1935, **27**, 2975).
Spanagel, Carothers, *J. Am. Chem. Soc.*, 1935, **57**, 931.
Paul, *Bull. soc. chim.*, 1934, **1**, 971.
Wojcik, Adams, *J. Am. Chem. Soc.*, 1933, **55**, 4941.
Bennett, Heathcoat, *J. Chem. Soc.*, 1929, 273.
Müller, Rölz, Gero, *Monatsh.*, 1928, **50**, 107.
v. Braun, Deutsch, Schmatloch, *Ber.*, 1912, **45**, 1250.
Hamonet, *Bull. soc. chim.*, 1905, **33**, 529.
v. Braun, Steindorff, *Ber.*, 1905, **38**, 959.
Lespieau, *Ann. chim. phys.*, 1912, **27**, 175.
Kaufman, Reeve, *Organic Syntheses*, 1946, XXVI, 83.

Pentamethyleneimine.
See Piperidine.

Pentamethylene iodide.
See 1 : 5-Di-iodo-*n*-pentane.

Pentamethylene oxide.
See Tetrahydropyran.

Pentamethylene sulphide (*Tetrahydrothiopyran*)



$C_5H_{10}S$ MW, 102

Found in crude Mexican petroleum. Cryst. M.p. 13°. B.p. 141°/755 mm. (141·5–142°/747 mm.). Sol. most org. solvents. Insol. H_2O. D^{15}_4 0·9889, D^{18}_4 0·9943, D^{20}_4 0·9849. n^{18}_D 1·5046.

Stable at 200°. Volatile in steam. Tetranitromethane in EtOH ⟶ golden-yellow col. Dil. HNO_3 ⟶ pentamethylene sulphoxide. $KMnO_4$ ⟶ pentamethylene sulphone.

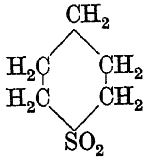
B,HgCl₂ : leaflets from EtOH. M.p. 137·5°. Sol. hot EtOH. Insol. H_2O, Et_2O.

Methiodide : needles from EtOH. Sublimes at 192° (162°) without melting. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O, C_6H_6, ligroin.

Methochloroplatinate : m.p. 225°. Sol. H_2O.

Clarke, *J. Chem. Soc.*, 1912, **101**, 1805.
v. Braun, Trümpler, *Ber.*, 1910, **43**, 548.
Grischkewitsch - Trochimowski, *Chem. Zentr.*, 1923, I, 1503.

Pentamethylene sulphone

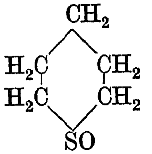


$C_5H_{10}O_2S$ MW, 134

Cryst. from H_2O. M.p. 98·5–99°. Sol. H_2O, EtOH. Less sol. Et_2O. Insol. ligroin.

Grischkewitsch - Trochimowski, *Chem. Zentr.*, 1923, I, 1503.

Pentamethylene sulphoxide



$C_5H_{10}OS$ MW, 118

Yellow liq. which solidifies to glassy mass in desiccator.

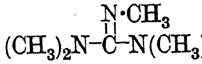
Grischkewitsch - Trochimowski, *Chem. Zentr.*, 1923, I, 1503.

Pentamethylene-tetrazole.
See Cardiazole.

Pentamethylene-urea.
See under Piperidine-*N*-carboxylic Acid.

Pentamethylene-urethane.
See under Piperidine-*N*-carboxylic Acid.

Pentamethylguanidine



$C_6H_{15}N_3$ MW, 129

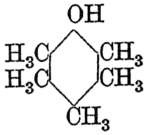
Liq. B.p. 155–60°. Sol. H_2O, org. solvents. Very hygroscopic. Fumes slightly in moist air. Readily absorbs CO_2. Strong base.

B,HAuCl₄ : needles from H_2O. M.p. 130–2°.

Picrate : yellow needles from H_2O. M.p. 165–6° decomp.

Schenck, *Z. physiol. Chem.*, 1912, **77**, 386.
Lecher, Graf, *Ber.*, 1923, **56**, 1329.

Pentamethylphenol (*Hydroxypentamethylbenzene*)



$C_{11}H_{16}O$ MW, 164

Needles from EtOH or pet. ether. M.p. 126°. B.p. 267°. Gives no col. with $FeCl_3$.

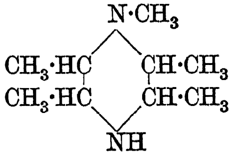
Me ether : pentamethylanisole. $C_{12}H_{18}O$. MW, 178. Needles from EtOH. M.p. 63–4°. (White plates from Et_2O. M.p. 119°). Sol. EtOH.

Benzoyl : rhombic plates from EtOH. M.p. 127°.

Phenylurethane : needles from EtOH–pet. ether. M.p. 215°.

Hey, *J. Chem. Soc.*, 1931, 1593.
Dimroth, Leichtlin, Friedemann, *Ber.*, 1917, **50**, 1543.
Hofmann, *Ber.*, 1885, **18**, 1826.
Kolka, Vogt, *J. Am. Chem. Soc.*, 1939, **61**, 1464.

2 : 3 : 4 : 5 : 6-Pentamethylpiperazine
(2 : 3 : 4 : 5 : 6-*Pentamethylhexahydropyrazine*)



$C_9H_{20}N_2$ MW, 156

γ-Form :

Prisms. M.p. 45°. B.p. 201–2°. Very hygroscopic.

B,H₂O : prisms from Me_2CO. M.p. 73–4°. Sol. EtOH, AcOEt. Spar. sol. C_6H_6, Et_2O.

B,2HCl : needles. M.p. about 300°. Sol. H_2O. Insol. EtOH.

B,HI : prisms from H_2O or EtOH. M.p. 161–2°. Very sol. H_2O, hot EtOH.

B,2HI : prisms from EtOH. M.p. 240° decomp. Sol. H_2O, EtOH, MeOH. Almost insol. Me_2CO, AcOEt.

N-p-*Toluenesulphonyl* : prisms from EtOH, needles from pet. ether. M.p. 124°. *B,HCl* : prisms from EtOH. M.p. 250°. d-*Tartrate* : needles from Me_2CO.Aq. M.p. 174–5°. $[\alpha]_{5461}$ + 9·7° in H_2O. d-*Camphor*-10-*sulphonate* : prisms from H_2O. M.p. 208–11°. $[\alpha]_{5461}$ + 28·4° in $CHCl_3$.

N-*Nitroso* : pale yellow oil. M.p. 24–5°, B.p. 155–7°/15 mm. Very sol. EtOH, Et_2O, CS_2, pet. ether. Less sol. hot than cold H_2O. d-*Camphor*-10-*sulphonate* : cryst. from Me_2CO.Aq. M.p. 213–15° decomp. $[\alpha]_{5461}$ + 15·2° in H_2O.

d-*Tartrate* : cryst. from MeOH. M.p. 164–5°. $[\alpha]_{5461}$ + 20·3° in H_2O.

Di-d-camphor-10-sulphonate: cryst. from EtOH. M.p. 261°. $[\alpha]_{5461}$ + 20·0° in H_2O.

Phenylthiourea: prisms from pet. ether. M.p. 154°. Sol. EtOH. C_6H_6. Spar. sol. pet. ether.

β-*Form*:

p-*Toluenesulphonyl*: prisms from EtOH or pet. ether. M.p. 100–1°.

Phenylthiourea: prisms from pet. ether. M.p. 95°.

Kipping, *J. Chem. Soc.*, 1932, 1340; 1933, 143.

Pentanal.

See Valeraldehyde.

1-Pentanalone-2.

See Propylglyoxal.

1-Pentanalone-4.

See Levulinic Aldehyde.

Pentandiol.

See Dihydroxypentane *and* Pentamethylene Glycol.

Pentandione-2 : 3.

See Acetylpropionyl.

Pentandione-2 : 4.

See Acetylacetone.

***n*-Pentane**

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

C_5H_{12} MW, 72

Found in American petroleum. F.p. — 129°. B.p. 36·00°. D_4^{20} 0·62632. n_D^{20} 1·35769. Sol. 200 parts H_2O at 16°.

Noller, *Organic Syntheses*, 1931, XI, 84.

Shepard, Henne, Midgley, *J. Am. Chem. Soc.*, 1931, **53**, 1948.

Pentane-1-carboxylic Acid.

See *n*-Caproic Acid.

Pentane-2-carboxylic Acid.

See 1-Methylvaleric Acid.

Pentane-3-carboxylic Acid.

See Diethylacetic Acid.

Pentane-1 : 1-dicarboxylic Acid.

See *n*-Butylmalonic Acid.

Pentane-1 : 2-dicarboxylic Acid.

See Propylsuccinic Acid.

Pentane-1 : 3-dicarboxylic Acid.

See 1-Ethylglutaric Acid.

Pentane-1 : 4-dicarboxylic Acid.

See 1-Methyladipic Acid.

Pentane-1 : 5-dicarboxylic Acid.

See Pimelic Acid.

Pentane-2 : 2-dicarboxylic Acid.

See Methylpropylmalonic Acid.

Pentane-2 : 3-dicarboxylic Acid.

See 1-Methyl-2-ethylsuccinic Acid.

Pentane-2 : 4-dicarboxylic Acid.

See 1 : 3-Dimethylglutaric Acid.

Pentane-3 : 3-dicarboxylic Acid.

See Diethylmalonic Acid.

Pentanol.

See *n*-Amyl Alcohol, Methyl-*n*-propylcarbinol *and* Diethylcarbinol.

Pentanolamine.

See Aminopentanol.

1-Pentanolone-2 (*Butyrylcarbinol, 2-keto-n-amyl alcohol*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH_2OH$

$C_5H_{10}O_2$ MW, 102

Oil. B.p. 54–6°/11 mm. (45°/12 mm.). D_4^{20} 0·9860. n_D^{20} 1·4234.

Me ether: $C_6H_{12}O_2$. MW, 116. B.p. 146–8° (152–3°/745 mm.), 117°/175 mm. D_4^{20} 0·9139. n_D^{20} 1·4119. *Semicarbazone*: needles. M.p. 97–8°. Very sol. H_2O, EtOH, Et_2O, AcOEt, C_6H_6. 2 : 4-*Dinitrophenylhydrazone*: m.p. 128·5–129°.

Et ether: $C_7H_{14}O_2$. MW, 130. B.p. 167°, 64–5°/17 mm., 60°/11 mm. D_4^{16} 0·9218. Spar. sol. H_2O. Reduces $NH_3.AgNO_3$. Forms bisulphite comp. *Semicarbazone*: m.p. 86–7°.

Osazone: m.p. 110–11°.

2 : 4-*Dinitrophenylosazone*: m.p. 234–6° decomp.

Henze, Rigler, *J. Am. Chem. Soc.*, 1934, **56**, 1350.

Maruyama, *Chem. Abstracts*, 1933, **27**, 1863.

Sommelet, *Bull. soc. chim.*, 1911, **9**, 36.

Schmidt, Ascherl, *Ber.*, 1925, **58**, 358.

1-Pentanolone-3 (*2-Propionylethyl alcohol, 3-keto-n-amyl alcohol*).

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2OH$

$C_5H_{10}O_2$ MW, 102

Me ether: b.p. 62–3°/24 mm. D_4^{20} 0·9162. n_D^{20} 1·4120. Does not form a semicarbazone.

Nazarov, Torgov, *Chem. Abstracts*, 1948, **42**, 7736.

1-Pentanolone-4.

See 3-Acetopropyl Alcohol.

2-Pentanolone-3 (*Methylpropionylcarbinol, 1-hydroxydiethyl ketone*)

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH(OH){\cdot}CH_3$

$C_5H_{10}O_2$ MW, 102

B.p. 152·5°/761 mm., 63°/20 mm., 45–8°/11 mm. D_4^{20} 0·9742. n_D^{20} 1·4218. Vigorously reduces $NH_3.AgNO_3$ and Fehling's.

Me ether: $C_6H_{12}O_2$. MW, 116. B.p. 133°/729 mm.

Et ether: $C_7H_{14}O_2$. MW, 130. B.p. 145°/727 mm.

Butyl ether: $C_9H_{18}O_2$. MW, 158. B.p. 80–2°/35 mm. D_4^{20} 0·8693. n_D^{20} 1·4143.

Semicarbazone: m.p. 208–9°.

Phenylhydrazone: needles from EtOH.Aq. M.p. 107°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6.

Osazone: m.p. 167°.

2 : 4 - *Dinitrophenylosazone*: cryst. from $PhNO_2$. M.p. 270–1° decomp.

Vénus-Daniloff, *Bull. soc. chim.*, 1928, **43**, 582.
Henze, Murchison, *J. Am. Chem. Soc.*, 1933, **55**, 4257.
Schmidt, Ascherl, *Ber.*, 1925, **58**, 357.
Gauthier, *Ann. chim. phys.*, 1909, **16**, 324; *Compt. rend.*, 1911, **152**, 1101.

2-Pentanolone-4.
See Acetoisopropyl Alcohol.

3-Pentanolone-2 (*Ethylacetylcarbinol*, 1-*acetopropyl alcohol, methyl* 1-*hydroxypropyl ketone*)

$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CO{\cdot}CH_3$

$C_5H_{10}O_2$ MW, 102

B.p. 147–8°/761 mm., 77°/35 mm., 59–59·5°/27 mm. D_4^{20} 0·9500. Misc. with H_2O, EtOH, and most other org. solvents.

Me ether: $C_6H_{12}O_2$. MW, 116. B.p. 154–5°/746 mm. n_D^{20} 0·8913. *Semicarbazone*: m.p. 120·5°.

Et ether: $C_7H_{14}O_2$. MW, 130. B.p. 76°/67 mm. D^{17} 0·8849. n^{14} 1·4075. *Semicarbazone*: m.p. 93–5°.

Acetyl: b.p. 68–70°/13 mm.

Semicarbazone: m.p. 216–17°.

Osazone: m.p. 167°.

2 : 4 - *Dinitrophenylosazone*: cryst. from $PhNO_2$. M.p. 270–1° decomp.

Grard, *Ann. chim.*, 1930, **13**, 336.
Vénus-Daniloff, *Bull. soc. chim.*, 1928, **43**, 582.
Bouis, *Bull. soc. chim.*, 1932, **51**, 1177.

Pentanone.
See Diethyl Ketone *and* Methyl propyl Ketone.

Pentantriol-1 : 2 : 3 (1-*Ethyl glycerol*, 1 : 2 : 3-*trihydroxypentane*)

$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CH(OH){\cdot}CH_2OH$

$C_5H_{12}O_3$ MW, 120

Syrup. B.p. 192°/63·3 mm. D_0^{34} 1·0851. Misc. with H_2O, EtOH. Sol. Et_2O. Sweet taste.

Tribenzoyl: m.p. 99–100°.

Wagner, *Ber.*, 1888, **21**, 3349.
Delaby, *Compt. rend.*, 1923, **176**, 589.

Pentantriol-1 : 2 : 4 (1 : 2 : 4-*Trihydroxypentane*)

$CH_3{\cdot}CH(OH){\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2OH$

$C_5H_{12}O_3$ MW, 120

Liq. B.p. 180°/27 mm. D_0^0 1·135, D_0^{22} 1·120.

Wagner, *Ber.*, 1888, **21**, 3351.

Pentantriol-1 : 2 : 5 (1 : 2 : 5-*Trihydroxypentane*)

$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2OH$

$C_5H_{12}O_3$ MW, 120

Liq. B.p. 190–1°/13 mm. D_{15}^{20} 1·136. n_D^{20} 1·42799.

1 : 5-*Di-Me ether*: $C_7H_{16}O_3$. MW, 148. B.p. 94–5°/13 mm. D_{15}^{14} 0·976. n_D^{14} 1·43336. p-*Nitrobenzoyl*: m.p. 194–5°.

5-*Me* 1-*Et ether*: $C_8H_{18}O_3$. MW, 162. B.p. 99°/13 mm. D_4^{17} 0·955.

Triacetyl: b.p. 173–6°/20 mm., 159°/9 mm. D_{15}^{15} 1·123, D_{15}^{22} 1·112. n_D^{15} 1·4402, n_D^{22} 1·4369.

Triphenylurethane: m.p. 92°.

Paul, *Ann. chim.*, 1932, **18**, 303; *Bull. soc. chim.*, 1933, **53**, 417.
Wilson, *J. Chem. Soc.*, 1945, 49.

Pentantriol-1 : 3 : 5 (1 : 3 : 5-*Trihydroxypentane*)

$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2OH$

$C_5H_{12}O_3$ MW, 120

Liq. B.p. 188–9°/11 mm.

Blanchard, Paul, *Compt. rend.*, 1935, **200**, 1414.

Pentantriol-2 : 3 : 4 (1 : 3-*Dimethylglycerol*, 2 : 3 : 4-*trihydroxypentane*)

$CH_3{\cdot}CH(OH){\cdot}CH(OH){\cdot}CH(OH){\cdot}CH_3$

$C_5H_{12}O_3$ MW, 120

B.p. 244–6°, 152–3°/19 mm. Sol. EtOH, H_2O. Bitter taste.

Triformyl: m.p. 85°. B.p. 130–4°/15 mm.

Triacetyl: needles from Et_2O. M.p. 121°. B.p. 241–3°.

Delaby, Morel, *Bull. soc. chim.*, 1926, **39**, 416.
Reif, *Ber.*, 1908, **41**, 2740.

Pentantrione-2 : 3 : 4 (2 : 3 : 4-*Triketopentane*)

$CH_3{\cdot}CO{\cdot}CO{\cdot}CO{\cdot}CH_3$

$C_5H_6O_3$ MW, 114

Orange-red oil. B.p. 65–70°/30 mm. Bitter taste. Absorbs H_2O from the air with formation of hydrate, m.p. 52°. Turns brown on standing. Reduces boiling $CuSO_4$.Aq. to Cu.

2-*Oxime*: needles from AcOEt–ligroin. M.p. 75°. Sol. H_2O, EtOH, AcOEt. Insol. ligroin.

2 : 3-*Dioxime*: plates from H_2O. M.p. 128° decomp. Sol. H_2O, EtOH. Spar. sol. Et_2O, $CHCl_3$. Conc. H_2SO_4 $\longrightarrow$ green sol.

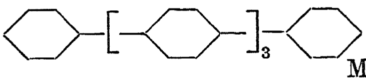
Semicarbazone: leaflets. M.p. 249°. Sol. AcOH. Spar sol. H_2O, EtOH, pet. ether. Alkalis $\longrightarrow$ yellow sols.

Phenylhydrazone: pale yellow leaflets. M.p. 249°. Sol. AcOH, Me_2CO, C_6H_6. Insol. pet. ether. Conc. H_2SO_4 $\longrightarrow$ yellow sol.

Di-phenylhydrazone: needles. M.p. 156°.

Wolff, *Ann.*, 1902, **325**, 139.
Sachs, Barschall, *Ber.*, 1901, **34**, 3052.

***p*-Pentaphenyl** (p-*Quinquiphenyl*)



$C_{30}H_{22}$ MW, 382

Needles from quinoline. M.p. 388·5°. Sublimes.

Gerngross, Dunkel, *Ber.*, 1924, **57**, 739.
Bursch, Weber, *J. prakt. Chem.*, 1936, **146**, 29.
Müller, Töpel, *Ber.*, 1939, **72**, 290.

Pentaphenylethane

$(C_6H_5)_3C{\cdot}CH(C_6H_5)_2$

$C_{32}H_{26}$ MW, 410

Plates from pet. ether. M.p. 166–78° in air, 182–5° in nitrogen. Sol. CS_2, C_6H_6. Spar. sol. Et_2O. Very spar. sol. EtOH, pet. ether. $CrO_3 \longrightarrow$ triphenylcarbinol + benzophenone.

Gomberg, Cone, *Ber.*, 1906, **39**, 1467.
Bachmann, *J. Am. Chem. Soc.*, 1933, **55**, 2135.

Pentaspadonic Acid.
See Pelandjauic Acid.

Pentatriacontane

$CH_3{\cdot}[CH_2]_{33}{\cdot}CH_3$

$C_{35}H_{72}$ MW, 492

Cryst. from warm EtOH. M.p. 75·0°. B.p. 331°/15 mm. $D_4^{74\cdot7}$ 0·7816. Spar. sol. boiling Et_2O.

Easterfield, Taylor, *J. Chem. Soc.*, 1911, **99**, 2305.
Clemmensen, *Ber.*, 1913, **46**, 1842.
Grün, Ulbrich, Krczil, *Z. angew. Chem.*, 1926, **39**, 421.

2-Pentenal (2-*Ethylacrylic aldehyde*)

$CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CHO$

C_5H_8O MW, 84

B.p. 124°/124 mm., 56°/65 mm. D^{21} 0·8532. n_D^{21} 1·4439.
Semicarbazone : m.p. 179°.

Prévost, *Bull. soc. chim.*, 1944, **11**, 218.

3-Pentenal (2-*Ethylidenepropionaldehyde*)

$CH_3{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CHO$

C_5H_8O MW, 84

B.p. 106°/760 mm., 39°/65 mm. D^{21} 0·8532. n_D^{21} 1·4209.
Semicarbazone : m.p. 123°.

Prévost, *Bull. soc. chim.*, 1944, **11**, 218.

4-Pentenal (*Allylacetaldehyde*)

$CH_2{:}CH{\cdot}CH_2{\cdot}CH_2{\cdot}CHO$

C_5H_8O MW, 84

B.p. 103–4°/749 mm. D_4^{20} 0·852. n_D^{20} 1·4191.
2 : 4-*Dinitrophenylhydrazone* : m.p. 120°.
Dimedone deriv. : m.p. 98°.

Hurd, Pollack, *J. Am. Chem. Soc.*, 1938, **60**, 1905.

1-Pentene (*Propylethylene, α-amylene, 1-pentylene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}CH_2$

C_5H_{10} MW, 70

F.p. −165°. B.p. 29·2°/739·7 mm. (39°). D_4^{20} 0·6411. n_D^{20} 1·3711. HI $\longrightarrow$ 2-iodopentane.

Adams, Kamm, Marvel, *J. Am. Chem. Soc.*, 1918, **40**, 1950.
Leendertse, Tulleners, Waterman, *Rec. trav. chim.*, 1934, **53**, 715.
Karanskii *et al.*, *Chem. Abstracts*, 1948, **42**, 2226.

2-Pentene (sym.-*Methylethylethylene, β-amylene, 2-pentylene*)

$CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH_3$

C_5H_{10} MW, 70

F.p. − 137°. B.p. 36·39°/760 mm. (36°/741 mm.). D_4^{20} 0·651, D_4^{25} 1·6437. n_D^{17} 1·3817, n_D^{20} 1·3308. HI $\longrightarrow$ 2-iodopentane.

Leendertse, Tulleners, Waterman, *Rec. trav. chim.*, 1934, **53**, 715.
Norris, Reuter, *J. Am. Chem. Soc.*, 1927, **49**, 2624.
Norris, *Organic Syntheses*, Collective Vol. I, 421.

Cis-isomer : f.p. − 151·37°. B.p. 36·08°/744 mm. D_4^{20} 0·6554. n_D^{20} 1·3828.
Trans-isomer : f.p. − 140·24°. B.p. 35·48°/744 mm. D_4^{20} 0·6475. n_D^{20} 1·3798.

Lucas, Schlatter, Jones, *J. Am. Chem. Soc.*, 1941, **63**, 27.

Pentene-carboxylic Acid.
See 2-Allylpropionic Acid, 3-Ethylidene-butyric Acid, Hydrosorbic Acid, 1-Methyl-2-ethylacrylic Acid, 1-Methyl-2-ethylidenepropionic Acid *and* Propylacrylic Acid.

Pentene-dial.
See Glutacondialdehyde.

1-Pentene-1 : 2-dicarboxylic Acid.
See Propylfumaric Acid *and* Propylmaleic Acid.

1-Pentene-1 : 3-dicarboxylic Acid.
See 1-Ethylglutaconic Acid.

2-Pentene-1 : 2-dicarboxylic Acid.
See Propylidene-succinic Acid.

2-Pentene-2 : 3-dicarboxylic Acid.
See dibasic-Hæmatinic Acid.

2-Pentene-3 : 4-dicarboxylic Acid.
See 1-Methyl-2-ethylidenesuccinic Acid.

2-Pentene-3 : 5-dicarboxylic Acid.
See 1-Ethylideneglutaric Acid.

2-Pentene oxide.
See sym.-Methylethylethylene oxide.

2-Pentene-2 : 3 : 5-tricarboxylic Acid.
See tribasic-Hæmatinic Acid.

Pentenic Acid.
See 2-Ethylidenepropionic Acid, 2-Ethylacrylic Acid *and* Allylacetic Acid.

Pentenol.
See Allylethyl Alcohol, Ethylvinylcarbinol, Methylallylcarbinol, Methylpropenylcarbinol *and* 2-Propylidene-ethyl Alcohol.

Pentenone.
See Ethylideneacetone, Ethyl vinyl Ketone *and* Methyl allyl Ketone.

2-Pentenylpenaldic Acid.
See Penaldic-F Acid.

2-Pentenylpenicillin.
See Penicillin-F.

2-Pentenylpenicilloic Acid.
See Penicilloic-F Acid.

2-Pentenylpenillamine.
See Penillamine-F.

2-Pentenylpenillic Acid.
See Penillic-F Acid.

2-Pentenylpenilloaldehyde.
See Penilloaldehyde-F.

3-Pentenyl phenyl Ketone.
See 4-Hexenophenone.

3-Penten-1-yne.
See Propenylacetylene.

1-Penten-3-yne (*Pirylene, methylvinylacetylene*)

$CH_3\cdot C⋮C\cdot CH⁚CH_2$

C_5H_6 MW, 66

B.p. 59·2°/760 mm. D_4^{20} 0·7401. n_D^{20} 1·4496.

Jacobson, Carothers, *J. Am. Chem. Soc.*, 1933, **55**, 1622.
Sargent, Buchman, Farquhar, *J. Am. Chem. Soc.*, 1942, **64**, 2692.

2-Penten-4-yn-1-ol (*γ-Hydroxypropenylacetylene*)

$HC⋮C\cdot CH⁚CH\cdot CH_2OH$

C_5H_6O MW, 82

B.p. 103°/100 mm., 90°/50 mm. n_D^{23} 1·4933.
α-Naphthylurethane : needles from pet. ether. M.p. 110–11°.

Heilbron, Jones, Lacey, McCombie, Raphael, *J. Chem. Soc.*, 1945, 77.
Haynes, Heilbron, Jones, Sondheimer, *J. Chem. Soc.*, 1947, 1583.

4-Penten-2-yn-1-ol (*Hydroxymethyl-vinylacetylene*)

$CH_2⁚CH\cdot C⋮C\cdot CH_2OH$

C_5H_6O MW, 82

B.p. 55–8°/10–11 mm. D_{20}^{20} 0·9598. n_D^{20} 1·4988.

Gverdtsiteli, *Chem. Abstracts*, 1948, **42**, 6738.

4-Penten-1-yn-3-ol (*Vinylethynylcarbinol*)

$CH_2⁚CH\cdot CH(OH)\cdot C⋮CH$

C_5H_6O MW, 82

B.p. 83·5–84·5°/150 mm. n_D^{16} 1·4545.
Phenylurethane : needles from pet. ether. M.p. 37°.
α-Naphthylurethane : needles from pet. ether. M.p. 127·5–128·5°.

Acetate : b.p. 87–8°/100 mm. n_D^{23} 1·4319.

Jones, McCombie, *J. Chem. Soc.*, 1942, 733.

Penthrit.
See under Pentaerythritol.

Pentocystine (4 : 4′-*Diamino*-4 : 4′-*dicarboxy*-1 : 1′-*dibutyl disulphide*)

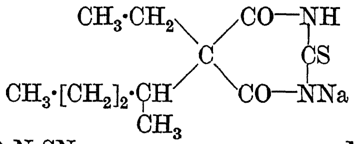
$S\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH(NH_2)\cdot COOH$
$S\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH(NH_2)\cdot COOH$

$C_{10}H_{20}O_4N_2S_2$ MW, 296

Needles from H_2O. Decomp. at 269–72°.
Diformyl : cryst. from H_2O. M.p. 120–2°.

du Vigneaud, Dyer, Jones, Patterson, *J. Biol. Chem.*, 1934, **106**, 403.

Pentothal (*Sodium* 5-*ethyl*-5-(1-*methylbutyl*) *thiobarbiturate*

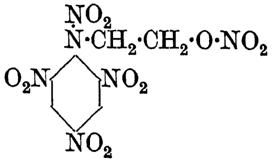


$C_{11}H_{17}O_2N_2SNa$ MW, 264

Anæsthetic.
Free acid : m.p. 158–9°.

Volwiler, Tabern, U.S.P.s 2,153,729, 2,153,731, (*Chem. Abstracts*, 1939, **33**, 5599).

Pentryl (β-N : 2 : 4 : 6-*Tetranitroanilinoethyl nitrate*)



$C_8H_6O_{11}N_6$ MW, 362

Cryst. from $CHCl_3$. M.p. 129°. High explosive.

Waldkötter, *Rec. trav. chim.*, 1938, **57**, 1294.

Pentylene.
See Pentene.

γ-Pentylene Glycol.
See 1 : 4-Dihydroxypentane.

γ-Pentylene oxide.
See 2-Methyltetrahydrofuran.

1-Pentyne (n-*Propylacetylene*)

$CH_3\cdot CH_2\cdot CH_2\cdot C⋮CH$

C_5H_8 MW, 68

Liq. F.p. −106° (−95°). B.p. 40° (48–9°). D_4^{20} 0·6908. n_D^{20} 1·3852.

Picon, *Compt. rend.*, 1914, **158**, 1346.
Bouis, *Ann. chim.*, 1928, **9**, 402.
Bourguel, *Ann. chim.*, 1925, **3**, 191.
Morehouse, Maass, *Chem. Abstracts*, 1935, **29**, 1059.
Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 484.

2-Pentyne (*Methylethylacetylene*)

$CH_3·CH_2·C⋮C·CH_3$

C_5H_8 MW, 68

Found in petroleum and fusel oil. F.p. — 101°. B.p. 55·5°/760 mm. $D_4^{17·2}$ 0·7127. $n_D^{17·2}$ 1·4045.

Faworsky, *J. prakt. Chem.*, 1888, **37**, 387.
v. Risseghem, *Compt. rend.*, 1914, **158**, 1696.
Scherrill, Launspach, *J. Am. Chem. Soc.*, 1938, **60**, 2562.

1-Pentyne-1-carboxylic Acid.
See Propylpropiolic Acid.

1-Pentyne-4-carboxylic Acid (2-*Acetylenyl-isobutyric acid*)

COOH
$CH_3·CH·CH_2·C⋮CH$

$C_6H_8O_2$ MW, 112

Oil. B.p. 207–8°/768 mm. Spar. sol. H_2O.
Et ester: $C_8H_{12}O_2$. MW, 140. Oil. B.p. 165–7°/757 mm. D_4^4 0·95424, D_{20}^{20} 0·93989. n_a^{17} 1·42654.

Perkin, Simonsen, *J. Chem. Soc.*, 1907, **91**, 832.
Gardner, Perkin, *ibid.*, 853.

1-Pentynol-3.
See Ethylethynylcarbinol.

1-Pentynol-4 (β-*Hydroxypropyl-acetylene*, 4-*hydroxypropylacetylene*)

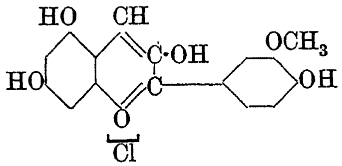
$CH_3·CH(OH)·CH_2·C⋮CH$

C_5H_8O MW, 84

B.p. 74·6°/100 mm. n_D^{16} 1·4406.

Haynes, Jones, *J. Chem. Soc.*, 1946, 954.

Peonidin chloride (3 : 5 : 7 : 4′-*Tetrahydroxy-3′-methoxyflavylium chloride*)

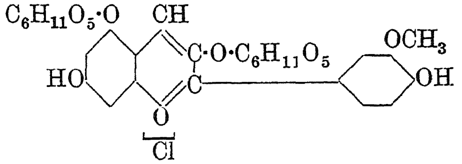


$C_{16}H_{13}O_6Cl$ MW, 336·5

Reddish-brown needles + H_2O from 20% HCl. Two modifications of a hydrate containing $1\frac{1}{2}H_2O$ have also been obtained. Sol. EtOH with violet-red col. Mod. sol. cold H_2O with brownish-red col. Spar. sol. dil. HCl.
5-*Benzoyl*: scarlet needles + $\frac{1}{2}H_2O$. Sol. MeOH, EtOH.

Nolan, Pratt, Robinson, *J. Chem. Soc.*, 1926, 1968.
Murakami, Robinson, *J. Chem. Soc.*, 1928, 1537.
Willstätter, Nolan, *Ann.*, 1915, **408**, 141.

Peonin chloride (7 : 4′-*Dihydroxy-3 : 5-di-β-glucosido-3′-methoxyflavylium chloride*)

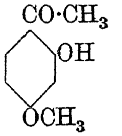


$C_{28}H_{33}O_{16}Cl$ MW, 660·5

Colouring matter of deep violet-red peonies. Reddish-violet needles + H_2O from aq. HCl. Decomp. at 165–7°. Hyd. ⟶ 2 mols. glucose + peonidin chloride.

Robinson, Todd, *J. Chem. Soc.*, 1932, 2493.
Willstätter, Nolan, *Ann.*, 1915, **408**, 136.

Peonol (2-*Hydroxy-4-methoxyacetophenone, resacetophenone 4-methyl ether, pæonol*)



$C_9H_{10}O_3$ MW, 166

Obtained from the root of *Paeonia Moutan* (China, Japan) and a constituent of several ethereal oils *e.g.* from *Paeonia arborea*, Don. Needles from EtOH. M.p. 50°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Volatile in steam. $D^{81·2}$ 1·1310. $n_D^{81·2}$ 1·54322. Gives reddish-violet col. with $FeCl_3$ in aq. or alc. solution. KOH fusion ⟶ resacetophenone, 2 : 4-dihydroxybenzoic acid, and resorcinol. HI ⟶ resacetophenone. Ac_2O ⟶ resacetophenone-4-methyl ether acetate. Br in AcOH ⟶ bromopeonol.
Me ether: 2 : 4-dimethoxyacetophenone. *See under* Resacetophenone.
Et ether: *see under* Resacetophenone.
Acetyl: needles from EtOH. M.p. 46·5°.
Oxime: needles. M.p. 130°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O, ligroin.
Hydrazone: plates from dil. EtOH. M.p. 73–5°.
Phenylhydrazone: needles from EtOH. M.p. 108°. Sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. EtOH, ligroin. Insol. cold aq. alkalis.
o-*Nitrophenylhydrazone*: deep red prisms from AcOH. M.p. 217°.
m-*Nitrophenylhydrazone*: red plates from AcOH. M.p. 197°.
p-*Nitrophenylhydrazone*: red cryst. from AcOH. M.p. 238–9° (235–6° decomp.).
2 : 4 : 6-*Tribromophenylhydrazone*: needles from EtOH. M.p. 162°. Insol. hot aq. alkalis.
p-*Thiocyanophenylhydrazone*: cryst. from EtOH. M.p. 157–158·5°.
Semicarbazone: needles from EtOH. M.p. 221–2°.

$HgCl_2$ complex: cryst. from H_2O. M.p. 127°.
Glucoside: glucopeonol. $C_{15}H_{20}O_8$. MW, 328. Cryst. M.p. 82–3°. *Tetra-acetyl*: m.p. 144–5°.

Tahara, *Ber.*, 1891, **24**, 2460.
Brüll, Friedländer, *Ber.*, 1897, **30**, 300.
Nagai, *Ber.*, 1891, **24**, 2847.
Adams, *J. Am. Chem. Soc.*, 1919, **41**, 260.
Lindemann, Könitzer, Romanoff, *Ann.*, 1927, **456**, 304.
Mauthner, *J. prakt. Chem.*, 1942, **160**, 33.

Perabrodil (*Diodone, diodrast, pyelosil, pylumbrin*).

Salt of 2 : 2′-dihydroxydiethylamine and 3 : 5-di-iodo-4-pyridone-*N*-acetic acid (q.v.). Used for evaluation of renal activity.

Talbot, *Chem. Abstracts*, 1942, **36**, 4840.

Peracetic Acid (*Acetyl hydroperoxide*)

$CH_3{\cdot}CO{\cdot}O{\cdot}OH$

$C_2H_4O_3$ MW, 76

Liq. F.p. + 0·1°. Sol. H_2O, EtOH, Et_2O, H_2SO_4. Explodes violently above 110°. Unpleasant odour. Ketene ⟶ diacetyl peroxide. Strong corrosive action on skin. Used as disinfectant.

Acetyl: *see* Acetyl peroxide.

Benzoyl: acetyl benzoyl peroxide. Needles from ligroin. M.p. 40–1°. 0·639 parts sol. in 1000 parts H_2O at 25°. Stable when pure. Decomp. when damp or in presence of EtOH, Et_2O or acids. Explodes with hot conc. H_2SO_4. Pure comp. has no oxidising action.

m-*Nitrobenzoyl*: needles from MeOH–ligroin. M.p. 68°. Explodes on heating.

Konsort f. elektrochem. Ind., D.R.P., 269,937, (*Chem. Zentr.*, 1914, I, 716); 272,738, (*Chem. Zentr.*, 1914, I, 1615).
D'Ans, Frey, *Ber.*, 1912, **45**, 1848.
D'Ans, D.R.P., 251,802, (*Chem. Zentr.*, 1912, II, 1413).
Nef, *Ann.*, 1897, **298**, 283.
Freer, Nouy, *Am. Chem. J.*, 1902, **27**, 172.
Smit, *Rev. trav. chim.*, 1930, **49**, 675.
Greenspan, *J. Am. Chem. Soc.*, 1946, **68**, 907.

Perbenzoic Acid (*Benzoyl hydroperoxide*)

$C_6H_5{\cdot}CO{\cdot}O{\cdot}OH$

$C_7H_6O_3$ MW, 138

Plates from pet. ether. M.p. 41–3°. B.p. 97–110°/13–15 mm. Sol. most org. solvents. Spar. sol. H_2O, pet. ether. Very volatile. Sublimes in desiccator. Volatile in steam. Strong oxidising agent.

Acetyl: *see under* Peracetic Acid.

Benzoyl: *see* Benzoyl peroxide.

Braun, *Organic Syntheses*, Collective Vol. I (2nd ed.), 431.
Tiffeneau, *Organic Syntheses*, Collective Vol. I, 422.
Hibbert, Burt, *J. Am. Chem. Soc.*, 1925, **47**, 2240.
Baeyer, Villiger, *Ber.*, 1900, **33**, 858.
v. Pechmann, Vanino, *Ber.*, 1894, **27**, 1511.
Gambarjan, *Ber.*, 1909, **42**, 4008.
Prileshajew, *Chem. Zentr.*, 1911, I, 1279.
Bergmann, Witte, D.R.P. 409,779, (*Chem. Zentr.*, 1925, I, 1911).

Perbromoacetone.
See Hexabromoacetone.

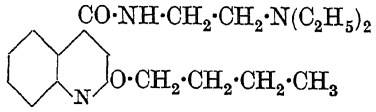
Perbromobenzoic Acid.
See Pentabromobenzoic Acid.

Perbromoethane.
See Hexabromoethane.

Perbromoethylene.
See Tetrabromoethylene.

Perbromoindone.
See Hexabromoindone-3.

Percaine (*Nupercaine*)



$C_{20}H_{29}O_2N_3$ MW, 343

Plates. M.p. 97–8°. Sol. H_2O, EtOH, C_6H_6. Insol. Et_2O. Aq. sol. shows blue fluor. Local anæsthetic about ten times more powerful than cocaine.

Miescher, *Helv. Chim. Acta*, 1932, **15**, 169.
Uhlmann, *Chem. Zentr.*, 1929, II, 1816.

Perchloroacetone.
See Hexachloroacetone.

Perchlorobenzene.
See Hexachlorobenzene.

Perchlorobenzoic Acid.
See Pentachlorobenzoic Acid.

Perchlorocyclopentene.
See Octachlorocyclopentene.

Perchloroethane.
See Hexachloroethane.

Perchloroethylene.
See Tetrachloroethylene.

Perchloroindone.
See Hexachloroindone-3.

Perchloromethyl Mercaptan (*Thiocarbonyl tetrachloride, trichloromethyl sulphochloride*)

$CCl_3{\cdot}SCl$

CCl_4S MW, 186

Pale yellow oil. B.p. 149° (146·5–148°), 73°/50 mm. $D_0^{12·8}$ 1·712, D_4^{11} 1·71785. n_D 1·54835. Decomp. above 200°. $SnCl_2$ ⟶

$CSCl_2 + SnCl_4$. $Fe + HCl \longrightarrow CSCl_2 + CCl_4$. $Fe \longrightarrow CCl_4$.

Helferich, Reid, *J. Am. Chem. Soc.*, 1921, **43**, 591.
Dyson, *Organic Syntheses*, Collective Vol. I, 493.

Pereirine

$C_{20}H_{26}ON_2$ MW, 310

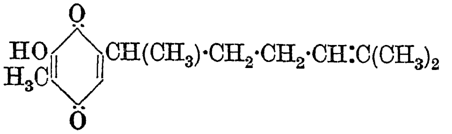
Pale yellow amorph. powder + $\frac{1}{2}H_2O$. M.p. 135° decomp. $[\alpha]_D$ + 137·5° in EtOH. Sol. EtOH, MeOH, Et_2O, $CHCl_3$, AcOEt, C_6H_6, acids. Insol. H_2O. Very bitter taste. Conc. H_2SO_4 ⟶ violet-red col.

Me ether: $C_{21}H_{28}ON_2$. MW, 324. Yellow amorph. powder. M.p. 195–7° decomp. Very sol. MeOH, EtOH, AcOEt. Insol. H_2O, Me_2CO, Et_2O, pet. ether.

Methiodide: yellow amorph. powder. M.p. 233–5° decomp. Sol. MeOH, EtOH. Spar. sol. H_2O. Insol. Et_2O, Me_2CO, $CHCl_3$.

Bertho, Moog, *Ann.*, 1934, **509**, 241.

Perezone (*Pipitzahoic acid*)



$C_{15}H_{20}O_3$ MW, 248

Found in rind of *Trixis pipitzhuac*. Golden-yellow leaflets from H_2O, plates from Et_2O. M.p. 104–6°. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Mod. sol. pet. ether. Insol. H_2O. $[\alpha]_D^{20}$ −17·0° in Et_2O. Volatile in steam. Racemises readily on sublimation. Alkalis ⟶ intense purple col.

Me ether: $C_{16}H_{22}O_3$. MW, 262. B.p. 128–33°/0·002 mm.

Fichter, Jetzer, Leepin, *Ann.*, 1913, **395**, 15.
Bayer, D.R.P., 278,090, (*Chem. Zentr.*, 1914, II, 900).
Kögl, Boer, *Rec. trav. chim.*, 1935, **54**, 785.

Perfluoropentane.

See Dodecafluoropentane.

Perfluorosuccinic Acid.

See Tetrafluorosuccinic Acid.

Performic Acid (*Formyl hydroperoxide*)

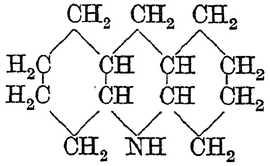
H·CO·O·OH

CH_2O_3 MW, 62

Only obtained 90% pure. Liq. Misc. with H_2O, EtOH, Et_2O. Mod. sol. $CHCl_3$, C_6H_6. More volatile than formic acid. Strong oxidising agent. Explodes on heating with metals and metallic oxides.

D'Ans, Kneips, *Ber.*, 1915, **48**, 1137.
Greenspan, *J. Am. Chem. Soc.*, 1946, **68**, 907.

Perhydroacridine (*Tetradecahydroacridine*)



$C_{13}H_{23}N$ MW, 193

Cryst. M.p. 80°. B.p. 140°/14 mm.

B,HCl: does not melt below 300°. Mod. sol. H_2O, EtOH.

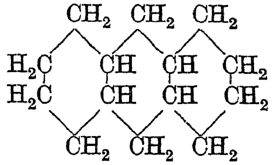
N-*Nitroso*: pale yellow. M.p. 217°.

Methiodide: m.p. 266°.

Picrate: cryst. M.p. 167°.

v. Braun, Petzold, Schultheiss, *Ber.*, 1923, **56**, 1349.

Perhydroanthracene (*Tetradecahydroanthracene*)



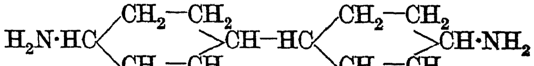
$C_{14}H_{24}$ MW, 192

Two isomeric compounds have been described.

1. Needles from MeOH. M.p. 61·5°.
2. Plates from EtOH. M.p. 93°. B.p. 128°/11 mm.

Fries, Schilling, *Ber.*, 1932, **65**, 1494.
Schroeter, *Ber.*, 1924, **57**, 1998.

Perhydrobenzidine (*Dodecahydrobenzidine, 4 : 4′-diaminodicyclohexyl*)



$C_{12}H_{24}N_2$ MW, 196

Cryst. M.p. 59°. B.p. 120°/0·8 mm. Turns yellowish-brown in air. Odour similar to piperidine. Weak base.

B,2HCl: amorph. powder. Decomp. at 290°.

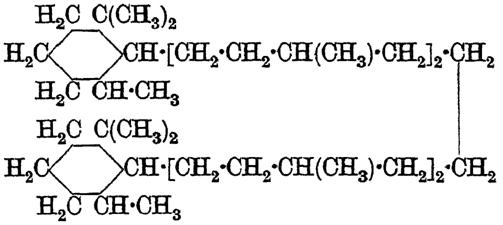
Dipicrate: red needles from EtOH. Decomp. explosively at 247°.

Balaš, Ševčenko, *Chem. Zentr.*, 1931, I. 3112.

Perhydrobixin.

See Bixane.

Perhydrocarotene



$C_{40}H_{78}$ MW, 558

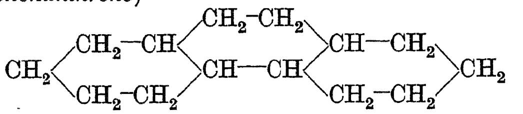
Needles from Et_2O. M.p. 65°. Sol. Et_2O, C_6H_6. Spar. sol. MeOH, EtOH. Conc. H_2SO_4 ⟶ blue sol.

Zechmeister, Cholnoky, Vrabély, *Ber.*, 1928, **61**, 566.

Perhydroindole.

See Octahydroindole.

Perhydrophenanthrene (*Tetradecahydrophenanthrene*)



$C_{14}H_{24}$ MW, 192

B.p. 86–9°/2 mm. D_4^{20} 0·9447. n_D^{20} 1·5011.

Pinkney *et al.*, *J. Am. Chem. Soc.*, 1936, **58**, 972.

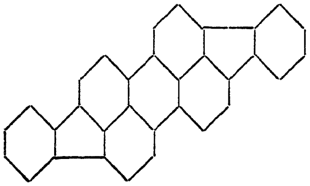
Peri-Acid.

See 1-Naphthylamine-8-sulphonic Acid.

β-Pericyclocamphane.

See Isobornylene.

Periflanthene

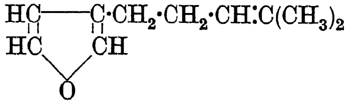


$C_{32}H_{16}$ MW, 400

Dark red solid. M.p. above 360°. Spar. sol. chlorobenzene, bromobenzene, phenol. Insol. Et_2O, xylene. Sublimes. Very stable and very resistant to oxidising agents. Sol. conc. H_2SO_4 at 100° with blue col.

Braun, Manz, *Ber.*, 1937, **70**, 1603.

Perillene

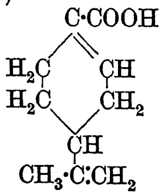


$C_{10}H_{14}O$ MW, 150

Found in oil from *Perilla citriodora*, Makino. B.p. 185–6°/760 mm., 93°/25 mm. D_4^{20} 0·9017. n_D^{21} 1·47053.

Kondo, Suzuki, *Ber.*, 1936, **69**, 2459.

Perillic Acid (*4-Isopropenylcyclohexene-1-carboxylic acid*)



$C_{10}H_{14}O_2$ MW, 166

l-.

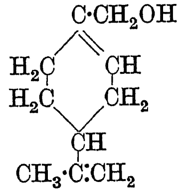
Leaflets from EtOH.Aq. M.p. 132–3° (130–1°). B.p. 164–5°/10 mm.

Amide: $C_{10}H_{15}ON$. MW, 165. Cryst. M.p. 164–5°.

Nitrile: $C_{10}H_{13}N$. MW, 147. B.p. 123°/15 mm., 116–18°/11 mm. D^{15} 0·9488, D^{20} 0·9439. n_D^{20} 1·4978.

Schimmel, *Chem. Zentr.*, 1910, II, 1758.
Semmler, Zaar, *Ber.*, 1911, **44**, 55.
Furukawa, Tomizawa, *Chem. Abstracts*, 1920, **14**, 2839.

Perillyl Alcohol ($\Delta^{1,8(9)}$-p-*Menthadienol-7, 1-hydroxymethyl-4-isopropenylcyclohexene*)



$C_{10}H_{16}O$ MW, 152

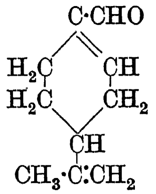
Found in ginger-grass oil. B.p. 118–21°/11 mm., 107–10°/12·5 mm. D^{20} 0·9690. n_D 1·4996. $[\alpha]_D$ − 7·0°. CrO_3 ⟶ perillyl aldehyde.

Acetyl: b.p. 123–6°/13 mm., 90–1°/4 mm. D^{20} 0·9785. n_D^{20} 1·47615.

Naphthylurethane: m.p. 146–7°.

Furukawa, Tomizawa, *Chem. Abstracts*, 1920, **14**, 2839.
Semmler, Zaar, *Ber.*, 1911, **44**, 54, 460.

Perillyl Aldehyde ($\Delta^{1,8(9)}$-p-*Menthadienal*)



$C_{10}H_{14}O$ MW, 150

l-.

Found in oil of *Perilla nankinensis*, D. B.p. 235–7°/750 mm., 104–5°/10 mm., 91°/4·5 mm. D^{20} 0·9645. n_D^{20} 1·5069. $[\alpha]_D^{20}$ − 145·8°. Reacts with Na_2SO_3 and $NaHSO_3$. Ag_2O ⟶ perillic acid.

syn-*Oxime*: prisms. M.p. 129°.

anti-*Oxime*: cryst. M.p. 102°. B.p. 147–8°/12 mm. Very sweet. *B,HCl*: m.p. 114°.

Semicarbazone: m.p. 199–200°.

Semioxamazone: m.p. 228°.

Phenylhydrazone: needles. M.p. 107·5°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 200–200·5°.

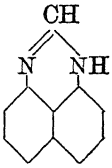
d-.

B.p. 234–6°/743 mm., 99–104°/9 mm. D^{15} 0·9730. n_D^{20} 1·5080. $[\alpha]_D$ + 137°. Reacts with Na_2SO_3 and $NaHSO_3$.

Oxime : m.p. 102°.
Phenylhydrazone : m.p. 107–8°.

Schimmel, *Chem. Zentr.*, 1910, II, 1758.
Semmler, Zaar, *Ber.*, 1911, **44**, 53, 460.
Furukawa, Tomizawa, *Chem. Abstracts*, 1920, **14**, 2839.
Hoshino, *ibid.*, 1410.

Perimidine (1 : 8-*Naphthiminazole*, peri-*naphthiminazole*)



$C_{11}H_8N_2$ MW, 168

Green cryst. from EtOH.Aq. M.p. about 222°. Sol. prac. all org. solvents, acids. Insol. H_2O.

B,HCl : yellowish-green needles from dil. HCl. Decomp. at 300°.

B,HNO₃ : long green needles from EtOH or hot H_2O.

Formate : cryst. M.p. 143°, solidifies and then remelts at 220°.

Acetate : cryst. from AcOH.Aq. M.p. 130°, solidifies and then remelts at 221°.

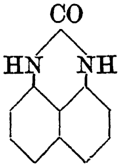
Oxalate : yellow cryst. from hot H_2O or EtOH. Decomp. at 270°.

Pyruvate : m.p. 207°. Very spar. sol. H_2O, EtOH.

Picrate : orange-red cryst. from EtOH. M.p. 249–50° decomp.

Sachs, *Ann.*, 1909, **365**, 83.

Perimidone



$C_{11}H_8ON_2$ MW, 184

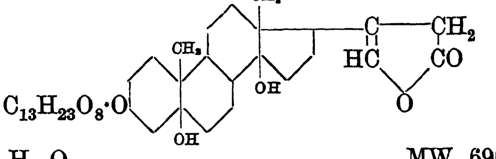
Cryst. from EtOH or C_6H_6. M.p. 304–5°. Spar. sol. most org. solvents.

Sachs, *Ann.*, 1909, **365**, 135.

Periodoacetone.
See Hexaiodoacetone.

Periodoethylene.
See Tetraiodoethylene.

Periplocin



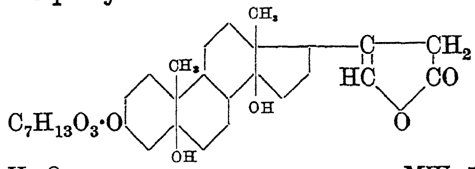
$C_{36}H_{56}O_{13}$ MW, 696

Cardiac glycoside from *Periploca graeca*, Linn. Needles from H_2O. M.p. 209° decomp. Sol. EtOH. Spar. sol. H_2O. Prac. insol. Et_2O, $CHCl_3$. $[\alpha]_D^{20}$ + 22·9° in MeOH. Hyd. ⟶ periplogenin + periplobiose. Enzyme hyd. ⟶ periplocymarin.

Tetra-acetyl : prisms from EtOH. M.p. 195°. Sol. EtOH, $CHCl_3$. Spar. sol. H_2O. $[\alpha]_D^{20}$ + 20·0° in EtOH.

Lehmann, *Arch. Pharm.*, 1897, **235**, 157.
Jacobs, Hoffmann, *J. Biol. Chem.*, 1928, **79**, 519.
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1193.

Periplocymarin

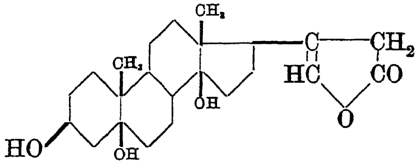


$C_{30}H_{46}O_8$ MW, 534

Prisms from MeOH.Aq. M.p. 212°. $[\alpha]_D^{20}$ + 29° in MeOH. HCl ⟶ periplogenin.

Jacobs, Hoffmann, *J. Biol. Chem.*, 1928, **79**, 519.
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1207.
Solacolu, Herrmann, *Compt. rend. soc. biol.*, 1934, **117**, 1138.
von Euw, Reichstein, *Helv. Chim. Acta*, 1948, **31**, 883.

Periplogenin



$C_{23}H_{34}O_5$ MW, 390

Aglycone of periplocin. Prisms from MeOH.Aq. M.p. 185°. $[\alpha]_D^{27}$ + 31·5° in EtOH.

Lehmann, *Arch. Pharm.*, 1897, **235**, 157.
Jacobs, Elderfield, *J. Biol. Chem.*, 1935, **108**, 497.
Kon, *J. Soc. Chem. Ind.*, 1934, **53**, 593.
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1193.

Perlatolic Acid

$C_{25}H_{32}O_7$ MW, 444

Needles from C_6H_6. M.p. 108°. Sol. EtOH, Et_2O, Me_2CO, C_6H_6. Spar. sol. pet. ether, ligroin. Sol. alkalis. Alc. $FeCl_3$ ⟶ violet col. $CHCl_3$ sol. + alkali ⟶ red col. on heating showing green fluor. on dilution with H_2O.

Di-Me ether-Me ester: $C_{28}H_{38}O_7$. MW, 486. Needles from MeOH. M.p. 57°.

Asahina, Fuzikawa, *Ber.*, 1935, **68**, 634.
Breadon, Keane, Nolan, *Chem. Abstracts*, 1942, **36**, 7068.

Perloline

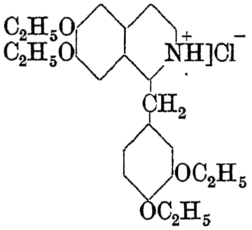
$C_{40}H_{34}O_7N_4$ MW, 682

Alkaloid from perennial rye grass. M.p. 181°. Sol. EtOH, $CHCl_3$. Mod. sol. Me_2CO, Et_2O, H_2O. Dil. sol. in $CHCl_3$ is golden yellow with green fluor.

Melville, Grimmett, *Nature*, 1941, **148**, 782.
Melville, Doak, *Chem. Abstracts*, 1943, **37**, 2075.
Grimmett, Melville, *Chem. Abstracts*, 1944, **38**, 1073.
White, Reifer, *Chem. Abstracts*, 1946, **40**, 1826.

Peropyrene.
See Dibenz-[cd, lm]-perylene.

Perparin (6 : 7-*Diethoxy*-1-[3′ : 4′-*diethoxybenzyl*]-*isoquinoline hydrochloride*)



$C_{24}H_{30}O_4NCl$ MW, 431·5

Cryst. M.p. 99–101°. Used as substitute for papaverine. Possesses stronger pharmacological properties.

Issekutz, Leinzinger, Dirner, *Chem. Abstracts*, 1932, **26**, 3836.
Pal, *ibid.*, 2510.

Perpropionic Acid (*Propionyl hydroperoxide*)

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}O{\cdot}OH$

$C_3H_6O_3$ MW, 90

Liq. F.p. − 13·5°. Explodes on heating $\longrightarrow CO_2 + CH_4 + C_2H_4 + C_2H_6$.

Propionyl: dipropionyl peroxide. Liq. Decomp. above 80°. Misc. with usual solvents.

Clover, Richmond, *Am. Chem. J.*, 1903, **29**, 191.
Konsort f. elektrochem. Ind., D.R.P., 269,937, (*Chem. Zentr.*, 1914, I, 716).
D'Ans, Frey, *Ber.*, 1912, **45**, 1850.
Fichter, Krummenacher, *Helv. Chim. Acta*, 1918, **1**, 146.

Perseitol.
See Persitol.

Perseulose

```
         OH   H    H    OH
HO·CH2—C————C————C————C—CO·CH2OH
         H    OH   OH   H
```

$C_7H_{14}O_7$ MW, 210

Needles from H_2O. M.p. 110–15° decomp. Sol. boiling EtOH. Sweet taste. Aq. sol. shows mutarotation. $[\alpha]_D^{20}$ − 90·0° ⟶ − 81·0° in H_2O. Reduces Fehling's.

Phenylosazone: needles from EtOH. M.p. 233°.

Bertrand, *Bull. soc. chim.*, 1909, **5**, 629.
La Forge, *J. Biol. Chem.*, 1916, **28**, 514.
Hann, Hudson, *J. Am. Chem. Soc.*, 1939, **61**, 336.
Wolfrom, Brown, Evans, *J. Am. Chem. Soc.*, 1943, **65**, 1021.

Persitol (*Perseitol*, d-α-*mannoheptitol*)

```
         OH   H    H    OH   OH
HO·CH2—C————C————C————C————C—CH2OH
         H    OH   OH   H    H
```

$C_7H_{16}O_7$ MW, 212

Obtained from seeds of *Persea gratissima* or *P. dymifolia*. Needles. M.p. 188°. Sol. H_2O, hot EtOH. Spar. sol. Et_2O. $[\alpha]_D^{20}$ + 4·53°. Oxidised by *B. xylinum* to perseulose.

Hepta-acetyl: m.p. 119°. Sol. EtOH. Insol. H_2O.

Heptanitrate: needles from EtOH. M.p. 138°.

Maquenne, *Ann. chim. phys.*, 1890, **19**, 5.
Fischer, *Ber.*, 1890, **23**, 936.
Bertrand, *Compt. rend.*, 1909, **149**, 226.
La Forge, *J. Biol. Chem.*, 1917, **28**, 520.
Ness, Hann, Hudson, *J. Am. Chem. Soc.*, 1948, **70**, 765.

Peruviol.
See Nerolidol.

Pervitin (2-*Methylamino*-1-*phenylpropane*, N : α - *dimethylphenylethylamine*, N - *methyl* - 1 - *phenylisopropylamine*)

$C_6H_5{\cdot}CH_2{\cdot}CH(CH_3){\cdot}NHCH_3$

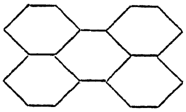
$C_{10}H_{15}N$ MW, 149

B.p. 78–80°/6 mm. Exhibits analeptic action. Excitatory and peripheral action similar to that of benzedrine..

B,HCl: cryst. from EtOH–Et_2O. M.p. 135–6°.

Woodruff, Lambooy, Burt, *J. Am. Chem. Soc.*, 1940, **62**, 922.

Perylene



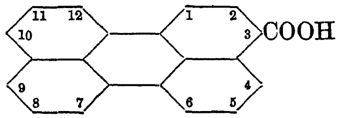
$C_{20}H_{12}$ MW, 252

Bronze plates from toluene or AcOH. M.p. 273–4° (264–5°). Sublimes at 350–400° in bronze leaflets. Sol. $CHCl_3$, CS_2. Less sol.

C_6H_6. Mod. sol. AcOH. Spar. sol. EtOH, Et_2O, Me_2CO. Insol. ligroin. Very dil. sols. show blue fluor. Conc. H_2SO_4 ⟶ deep green sol. which rapidly changes to bluish-green ⟶ blue ⟶ reddish-violet. CrO_3 in boiling AcOH ⟶ perylenequinone.

Scholl, Seer, Weitzenböck, *Ber.*, 1910, **43**, 2203.
Hansging, Zinke, *Monatsh.*, 1919, **40**, 404.
Marschalk, *Bull. soc. chim.*, 1928, **43**, 1388.
Zinke, Hauswirth, Blank, Grimm, *Monatsh.*, 1932, **61**, 1.
Clar, *Ber.*, 1932, **65**, 846.
Uchida, Takata, *J. Soc. Chem. Ind. Japan*, 1933, **36**, 222.
Morgan, Mitchell, *J. Chem. Soc.*, 1934, 536.

Perylene-3-carboxylic Acid

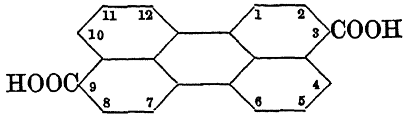


$C_{21}H_{12}O_2$ MW, 296

Yellowish-brown needles from $PhNO_2$. Does not melt below 300°. Sol. H_2SO_4 with violet col. and red fluor. Alkalis ⟶ yellow sols. with bluish-green fluor.
Me ester: m.p. 198–200°.
Et ester: m.p. 185–7°.
Cyclohexyl ester: m.p. 197–9°.
Phenyl ester: m.p. 237–40°.

I.G., F.P., 635,599, (*Chem. Zentr.*, 1929, I, 2472).
I.G., D.R.P. 703,500, (*Chem. Abstracts*, 1942, **36**, 781).

Perylene-3 : 9-dicarboxylic Acid



$C_{22}H_{12}O_4$ MW, 340

Orange micro-needles from $PhNO_2$. Sinters about 360°. Conc. H_2SO_4 ⟶ red sol. with orange-red fluor. Aq. alkalis ⟶ yellow sols. with bluish-green fluor. Spar. sol. org. solvents.
Di-Et ester: $C_{26}H_{28}O_4$. MW, 396. Yellow leaflets from xylene. M.p. 264–5°. Spar. sol. EtOH, toluene, xylene. Sols. are yellow with green fluor.
Dichloride: $C_{22}H_{10}O_2Cl_2$. MW, 377. Red needles from $PhNO_2$. Sol. C_6H_6, toluene, xylene. Sols. are yellow with yellowish-green fluor.
Dinitrile: $C_{22}H_{10}N_2$. MW, 302. Brown cryst. from $PhNO_2$. Does not melt below 360°. Spar. sol. EtOH, AcOH, xylene.

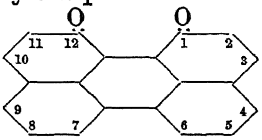
Pongratz, *Monatsh.*, 1927, **48**, 585; 1929, **52**, 9.
Pongratz, Griengl, Cecelsky, *Monatsh.*, 1933, **62**, 71.

Perylene-3 : 10-dicarboxylic Acid.

Reddish-brown needles from $PhNO_2$. Sol. 1000 parts hot $PhNO_2$. Conc. H_2SO_4 ⟶ reddish-violet col. slowly changing to brownish-red. Aq. alkalis ⟶ yellow sols. with strong green fluor.
Di-Et ester: red leaflets from xylene. M.p. 247–8°.
Dichloride: reddish-brown needles from benzoyl chloride.
Dinitrile: brown needles from aniline. M.p. 368–9° (sealed tube). Sol. $PhNO_2$, aniline. Spar. sol. AcOH, xylene with intense green fluor. Conc. H_2SO_4 ⟶ reddish-brown col.

Weitzenböck, Seer, *Ber.*, 1913, **46**, 1999.
Funke, *Monatsh.*, 1932, **59**, 193.

1 : 12-Perylenequinone



$C_{20}H_{10}O_2$ MW, 282

Reddish-brown needles from C_6H_6. M.p. 287°. Sol. hot AcOH, Py, $PhNO_2$, aniline, chlorobenzene. Spar. sol. hot EtOH, Me_2CO, CCl_4, C_6H_6, toluene. Conc. H_2SO_4 ⟶ brown sol. Insol. NaOH.

Zinke, Hanselmayer, *Monatsh.*, 1924, **45**, 232.

3 : 9-Perylenequinone.

Violet needles from $PhNO_2$. Reduced by hydrosulphite to red " vat " with intense green fluor.

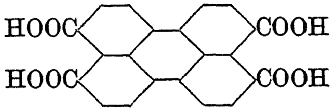
Zinke, Hirsch, *Monatsh.*, 1929, **52**, 18.

3 : 10-Perylenequinone.

Yellow needles from Py. Decomp. slowly above 350°. Sol. boiling AcOH, Py, $PhNO_2$, aniline, xylene, quinoline. Conc. H_2SO_4 ⟶ blood-red sol. with intense fluor.

Zincke, Unterkreuter, *Monatsh.*, 1919, **40**, 407.
Marschalk, *Bull. soc. chim.*, 1927, **41**, 74.

Perylene - 3 : 4 : 9 : 10 - tetracarboxylic Acid



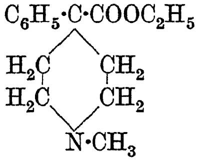
$C_{24}H_{12}O_8$ MW, 428

Brownish-red needles. Hot alkalis ⟶ brownish-yellow sols. with deep green fluor. Conc. H_2SO_4 ⟶ red sol. with orange-red fluor. Dry dist. of Ca salt ⟶ perylene.

Monoimide: brownish-red powder. Very dil. alkalis ⟶ deep red sols.

Kalle, D.R.P., 394,794, (*Chem. Zentr.*, 1924, II, 1276); 408,513, (*Chem. Zentr.*, 1925, I, 1811); 412,122, (*Chem. Zentr.*, 1925, I, 2666).

Pethidine (*Demerol, Dolantin, Isonipecaine, 1-methyl-4-phenylisonipecotic ethyl ester*)



$C_{15}H_{21}O_2N$ MW, 247

M.p. 30°. B.p. 155°/5 mm., 138°/3 mm., 115°/0·2 mm. Potent analgesic. Administered in the form of a salt, usually the hydrochloride (m.p. 187–9°).

Picrate: m.p. 189–90°.

Eisleb, *Ber.*, 1941, **74**, 1433.
Walton, Green, *J. Chem. Soc.*, 1945, 315.
Bergel, Morrison, Rinderknecht, *J. Chem. Soc.*, 1944, 265.
Battermann, Himmelsbach, *Journal of the American Medical Association*, 1943, **122**, 222.

Petroselaidic Acid.
See under Petroselic Acid.

Petroselic Acid (*Petroselinic acid, 5-heptadecylene-1-carboxylic acid, Δ^5-octadecylenic acid*)

$CH_3 \cdot [CH_2]_{10} \cdot CH{:}CH \cdot [CH_2]_4 \cdot COOH$

$C_{18}H_{34}O_2$ MW, 282

Cis:
Found in seeds of *Petroselinum sativum*. Leaflets. F.p. 27°. M.p. 33–4° (30°). B.p. 237–8°/18 mm. D^{40} 0·8700. n_D^{40} 1·4533. Dil. HNO_3 ⟶ *trans* acid. Ozone ⟶ adipic aldehyde + lauric acid.

Me ester: b.p. 208–10°/10 mm. D_4^{20} 0·8767. n_D^{20} 1·4501.

Amide: $C_{18}H_{35}ON$. MW, 281. Needles. M.p. 76°.

Triglyceride: tripetroselin. Cryst. F.p. 16·5°. M.p. 32° (26·4°). n_D^{40} 1·4619.

Trans: Petroselaidic acid.
Prisms. M.p. 54°.

Palazzo, Tamburello, *Atti accad. Lincei*, 1914, **23**, II, 352.
Vongerichten, Köhler, *Ber.*, 1909, **42**, 1638.
Steger, Loon, *Rec. trav. chim.*, 1927, **46**, 703.
Hilditch, Jones, *J. Soc. Chem. Ind.*, 1927, **46**, 174T.
Eibner, Widenmeyer, Schild, *Chem. Abstracts*, 1928, **22**, 1487.
Chernoyarova, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 149, (*Chem. Abstracts*, 1939, **33**, 6239).
Lumb, Smith, *J. Chem. Soc.*, 1952, 5032.

Petroselinic Acid.
See Petroselic Acid.

Petunidin (**chloride**)

$C_{16}H_{13}O_7Cl$ MW, 352·5

Forms several hydrates of different cryst. forms and solubilities depending on solvent used. Very similar in properties to myrtillidin chloride.

Willstätter, Burdick, *Ann.*, 1916, **412**, 224.
Cf. Bell, Robinson, *J. Chem. Soc.*, 1934, 1604.

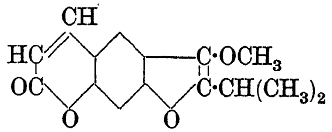
Petunin (**chloride**)

$C_{28}H_{33}O_{17}Cl$ MW, 676·5

Plates + $2H_2O$. M.p. about 179°. Cryst. are violet by transmitted light, and show copper lustre by reflected light. Sol. MeOH. MeOH+ $FeCl_3$ ⟶ violet col. Na_2CO_3.Aq. ⟶ violet sol. ⟶ blue on standing. 20% HCl ⟶ petunidin chloride + glucose.

Willstätter, Burdick, *Ann.*, 1916, **412**, 217.

Peucedanine



$C_{15}H_{14}O_4$ MW, 258

Found in roots of *Peucedanum officinale*. Prisms or plates. M.p. 109° (76°). Very sol. $CHCl_3$, CS_2. Sol. hot EtOH, Et_2O, AcOH. Spar. sol. C_6H_6, pet. ether. Insol. H_2O. HNO_3 ⟶ styphnic acid. Tasteless. Toxic to fish.

Heut, *Ann.*, 1875, **176**, 71.
Jassoy, Haensel, *Arch. Pharm.*, 1898, **236**, 668.
Späth, Klager, *Ber.*, 1933, **66**, 749.
Späth, Klager, Schlösser, *Ber.*, 1931, **64**, 2203.
Späth, Dobrovolny, *Ber.*, 1939, **72**, 52.

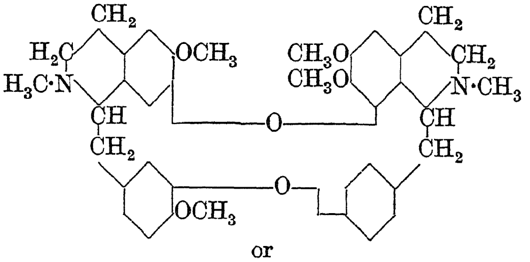
Peucenin

$C_{15}H_{16}O_4$ MW, 260

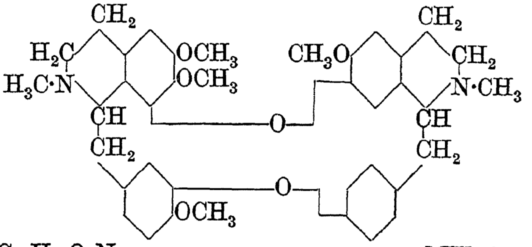
From the rhizome of *Peucedamum ostruthium*, Koch. M.p. 212°. Very sol. Et_2O, and MeOH. Spar sol. pet. ether, and H_2O.

Späth, Eiter, *Ber.*, 1941, **74**, 1851.

Phaeanthine (l-*Tetrandrine*)
Constitution represented by



or



$C_{38}H_{42}O_6N_2$ MW, 622

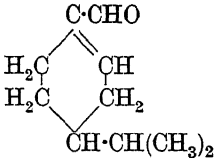
Alkaloid extracted from *Stephania cepharantha*, *Coceulus lavrifolius*, and *Phaeanthus ebracteolatus*. Hexagonal prisms from Et_2O. M.p. 216–17°. Sol. Et_2O, $CHCl_3$. Mod. sol. MeOH, Me_2CO. Spar. sol. EtOH. $[\alpha]_D^{16}$ — 285·5° in $CHCl_3$.

B,2HI : cryst. Decomp. at 268°.
B,2CH$_3$I : decomp. at 265°.
Picrate : decomp. at 263°.
Chloroaurate : m.p. 182–5°.

Kondo, Keimatsu, *Ber.*, 1935, **68**, 1505.
Chuang, Hsing, Kao, Chang, *Ber.*, 1939, **72**, 519.

Phaseolunatin.
See Linamarin.

Phellandral (Δ^1-*Tetrahydrocuminaldehyde*)



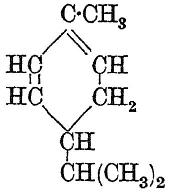
$C_{10}H_{16}O$ MW, 152

Occurs in water-fennel oil. B.p. 220–30°, 89°/5 mm. Mod. sol. H_2O. D^{20} 0·93. n_D^{20} 1·4911 (1·4903). Ox. ⟶ tetrahydrocuminic acid.

Oxime : m.p. 87–8°.
Semicarbazone : m.p. 204–5°.
p-*Nitrophenylhydrazone* : m.p. 169–70°.
2 : 4-*Dinitrophenylhydrazone* : orange-red needles from $CHCl_3$–EtOH. M.p. 202–3°.

Macbeth, Price, *J. Chem. Soc.*, 1935, 152.
Wallach, *Ann.*, 1905, **340**, 13.
Burger, Macbeth, *J. Chem. Soc.*, 1946, 145.

α-Phellandrene ($\Delta^{1,5}$-p-*Menthadiene*, 1-*methyl-4-isopropylcyclohexadiene*-1 : 5)



$C_{10}H_{16}$ MW, 136

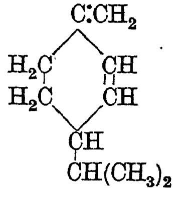
d-.
Occurs in bitter-fennel, elemi, and ginger-grass oils. B.p. 175–6°, 66–8°/16 mm., 66°/14 mm., 61°/11 mm. D_4^{25} 0·8463. n_D^{20} 1·4777.
α-*Nitrosite* : m.p. 113–14° (rapid heat). $[\alpha]_D^{20}$ — 141° in AcOEt.
β-*Nitrosite* : m.p. 105°. $[\alpha]_D^{19}$ + 45·8° in $CHCl_3$.

l-.
Occurs in oils of pimento, bay, and *Eucalyptus phellandra*, Smith. B.p. 174–7°/759 mm., 171–2°/758·2 mm., 67–8°/22 mm., 58–9°/16 mm. (60–60·5°/15 mm.). D_4^{20} 0·8425. n_D^{20} 1·4732. $[\alpha]_D^{20}$ — 112·76°.
α-*Nitrosite* : m.p. 120–1°.
β-*Nitrosite* : m.p. 105–6°.

dl-.
B.p. 175–6° decomp., 63–5°/15·5 mm. $n_D^{19.5}$ 1·4772.

Read, Storey, *J. Chem. Soc.*, 1930, 2781.
Galloway, Dewar, Read, *J. Chem. Soc.*, 1936, 1597.

β-Phellandrene ($\Delta^{2,1(7)}$-p-*Menthadiene*)



$C_{10}H_{16}$ MW, 136

d-.
Occurs in oil of *Bupleurum fructicosum*, Linn. B.p. 171–2°/766 mm., 57°/11 mm. D^{20} 0·8520. n_D^{20} 1·4788.
α-*Nitrosochloride* : m.p. 101–2°. $[\alpha]_D$ — 175° in $CHCl_3$.
β-*Nitrosochloride* : m.p. 100–1°. $[\alpha]_D$ — 285° in $CHCl_3$.
α-*Nitrosite* : m.p. 102°. $[\alpha]_D^{18.5}$ — 159·3° in $CHCl_3$.
β-*Nitrosite* : m.p. 97–8°. $[\alpha]_D$ — 159° in $CHCl_3$.

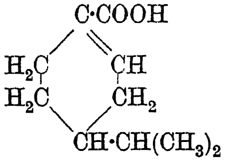
l-.
Occurs in oil of *Abies balsamea*. B.p. 172–4°/760 mm. $[\alpha]_D$ — 74·4°. $D_{15.5}^{15.5}$ 0·843. n_D^{20} 1·4826.
Tetrabromide : cryst. from AcOEt. M.p. 118–19°. $[\alpha]_D$ + 53°.

α-Nitrosochloride: m.p. 109°.

Francesconi, Sernagiotto, *Gazz. chim. ital.*, 1916, **46**, i, 119.
Wallach, *Ann.*, 1905, **340**, 1.
Berry, *Chem. Abstracts*, 1948, **42**, 2247.

α-Phellandrene Hydrate.
See Δ^2-*p*-Menthenol-1.

Phellandric Acid (3 : 4 : 5 : 6-*Tetrahydro*-p-*isopropylbenzoic acid*, 3 : 4 : 5 : 6-*tetrahydrocuminic acid*)



$C_{10}H_{16}O_2$ MW, 168

d-.
Cryst. from EtOH.Aq. M.p. 144–5°. $[\alpha]_D^{20}$ + 112·8° in MeOH.
p-*Chlorophenacyl ester*: needles from EtOH.Aq. M.p. 78–78·5°. $[\alpha]_D^{20}$ + 71° in $CHCl_3$.
p-*Bromophenacyl ester*: needles from EtOH.Aq. M.p. 86°. $[\alpha]_D^{20}$ + 68·1° in $CHCl_3$.

l-.
M.p. 144–5°. $[\alpha]_D^{20}$ − 112·6° in MeOH.
p-*Chlorophenacyl ester*: needles from EtOH.Aq. M.p. 78–78·5°. $[\alpha]_D^{20}$ − 57° in $CHCl_3$.
p-*Bromophenacyl ester*: needles from EtOH.Aq. M.p. 86°. $[\alpha]_D^{20}$ − 52·2° in $CHCl_3$.
p-*Nitrobenzyl ester*: pale yellow needles from MeOH. M.p. 56–7°.

dl-.
Cryst. from MeOH.Aq. M.p. 143–4°.
p-*Bromophenacyl ester*: m.p. 86–86·5°.

Cooke, Macbeth, Swanson, *J. Chem. Soc.*, 1940, 808.

Phellogenic Acid.
See Japanic Acid.

Phellonic Acid (1-*Hydroxybehenic acid*)

$CH_3 \cdot [CH_2]_{19} \cdot CH(OH) \cdot COOH$

$C_{22}H_{44}O_3$ MW, 356

Cryst. from $CHCl_3$. M.p. 96°.
Et ester: $C_{24}H_{48}O_3$. MW, 384. M.p. 70–1°.
Acetyl: m.p. 79°.
Et ether: $C_{24}H_{48}O_3$. MW, 384. M.p. 60°.

Fileti, *Gazz. chim. ital.*, 1897, **27**, ii, 300.
Zetzsche, Bähler, *Helv. Chim. Acta*, 1931, **14**, 642.

Phemitone.
See Prominal.

Phenacaine.
See under Holocaine.

Phenacetin.
See under *p*-Phenetidine.

Phenaceturic Acid (*Phenylacetylglycine*)

$C_6H_5 \cdot CH_2 \cdot CO \cdot NH \cdot CH_2 \cdot COOH$

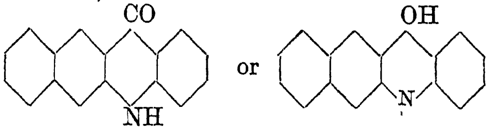
$C_{10}H_{11}O_3N$ MW, 193

Occurs in horse and dog urine. Leaflets from EtOH. M.p. 143° (136°). Sol. EtOH. Spar. sol. H_2O, Et_2O, hot C_6H_6. Hot HCl ⟶ glycine + phenylacetic acid.
Me ester: $C_{11}H_{13}O_3N$. MW, 207. Cryst. from EtOH. M.p. 86·5°. Sol. Et_2O, C_6H_6, $CHCl_3$, hot EtOH.
Et ester: $C_{12}H_{15}O_3N$. MW, 221. Prisms from EtOH. M.p. 82° (79°). Sol. hot EtOH.
Propyl ester: $C_{13}H_{17}O_3N$. MW, 235. Leaflets from H_2O. M.p. 31°.
Amide: $C_{10}H_{12}O_2N_2$. MW, 192. Plates from EtOH.Aq. M.p. 176–7°. Sol. hot H_2O. Insol. EtOH, Et_2O.
Nitrile: $C_{10}H_{10}ON_2$. MW, 174. Needles. M.p. 90·5°. Spar. sol. hot H_2O.
Hydrazide: m.p. 144–5°.
Azide: cryst. from Et_2O. M.p. 85–6°.

Hotter, *J. prakt. Chem.*, 1888, **38**, 98.

Phenacridol.
See Phenacridone.

Phenacridone (*Phenacridol, benzacridone, benzacridol*)



$C_{17}H_{11}ON$ MW, 245

Yellow leaflets from AcOH. M.p. 304–5°. Spar. sol. ord. org. solvents. Sols. show green fluor.
N-*Et*: $C_{19}H_{15}ON$. MW, 273. Needles from EtOH. M.p. 174–5°.
N-*Benzyl*: $C_{24}H_{17}ON$. MW, 335. Yellow needles from EtOH. M.p. 188–9°.

Schöpff, *Ber.*, 1893, **26**, 2590.

Phenacylacetone (*Acetophenoneacetone*, 1-*acetyl*-2-*benzoylethane*, α : δ-*diketo*-n-*amylbenzene*, ω-*acetonylacetophenone*)

$C_6H_5 \cdot CO \cdot CH_2 \cdot CH_2 \cdot CO \cdot CH_3$

$C_{11}H_{12}O_2$ MW, 176

Yellow oil. B.p. 161°/12 mm. Sol. hot H_2O. Insol. alkalis. No col. with $FeCl_3$.
Monoxime: m.p. 123°.
Dioxime: m.p. 108°.
Mono-semicarbazone: m.p. 191°.

Borsche, Fels, *Ber.*, 1906, **39**, 1926.
See also Finzi, *Gazz. chim. ital.*, 1912, **42**, ii, 356.

Phenacyl Alcohol (*Acetophenone alcohol benzoylcarbinol*, ω-*hydroxyacetophenone*)

$C_6H_5 \cdot CO \cdot CH_2OH$

$C_8H_8O_2$ MW, 136

Prisms from ligroin, plates from EtOH. M.p. anhyd. 86–7° (73–4° hydrated). B.p. 118–20°/11 mm. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. ligroin, hot H_2O. Sublimes at 56°/1 mm.

Me ether: ω-methoxyacetophenone. $C_9H_{10}O_2$. MW, 150. M.p. 7–7·5°. B.p. 228–30°, 118–20°/15 mm. *Semicarbazone*: m.p. 129°. 2:4-*Dinitrophenylhydrazone*: m.p. 191–2°.
Et ether: $C_{10}H_{12}O_2$. MW, 164. M.p. 81°. B.p. 134–6°/21 mm. *Oxime*: m.p. 55°.
Phenyl ether: $C_{14}H_{12}O_2$. MW, 212. M.p. 78°. B.p. 255–7°. Volatile in steam. *Oxime*: m.p. 113–14°.
o-*Nitrophenyl ether*: m.p. 118°.
p-*Nitrophenyl ether*: m.p. 144°.
o-*Tolyl ether*: m.p. 84°.
p-*Tolyl ether*: m.p. 68°.
Oxime: m.p. 70°.
Acetyl: m.p. 49° (40°). B.p. 270°.
Benzoyl: m.p. 118·5°. *Oxime*: m.p. 92°.
o-*Nitrobenzoyl*: m.p. 124·5°.
m-*Nitrobenzoyl*: m.p. 104·5°.
p-*Nitrobenzoyl*: m.p. 128·4°.

Rather, Reid, *J. Am. Chem. Soc.*, 1919, **41**, 79.
Wolff, *Ann.*, 1912, **394**, 42.
Dakin, *J. Biol. Chem.*, 1914, **18**, 92.
Allen, Scarrow, *Chem. Zentr.*, 1935, I, 1870.
Hann, Reid, Jamieson, *J. Am. Chem. Soc.*, 1930, **52**, 818.
Kelly, Kleff, *J. Am. Chem. Soc.*, 1932, **54**, 4444.

Phenacyl Aldehyde.
See Benzoylacetaldehyde.

Phenacylamine (*Phenomydrol, ω-aminoacetophenone, benzoylmethylamine*)

$C_6H_5{\cdot}CO{\cdot}CH_2NH_2$

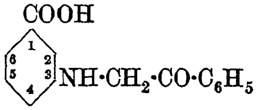
C_8H_9ON MW, 135

B,HCl: m.p. 186–7° decomp. (183–4°).
B,HBr: m.p. 217–18° decomp.
Sulphate: m.p. 182°.
N-*Benzoyl*: m.p. 126°.
N-p-*Toluenesulphonyl*: m.p. 79° decomp.
Oxime: m.p. 140°.
Picrate: m.p. 175–6° decomp.

Kindler, D.R.P., 571,795, (*Chem. Abstracts*, 1933, **27**, 4245).
Neber, Huh, *Ann.*, 1935, **515**, 292.
Neber, F.P., 768,604, (*Chem. Abstracts*, 1935, **29**, 475).
Tiffeneau, Orekoff, Roger, *Bull. soc. chim.*, 1931, **49**, 1761.

Phenacyl-*o*-aminobenzoic Acid.
See Phenacylanthranilic Acid.

Phenacyl-*m*-aminobenzoic Acid



$C_{15}H_{13}O_3N$ MW, 255

Cryst. from EtOH. M.p. 202°. Sol. AcOH, Py.
N-*Acetyl*: m.p. 217°.

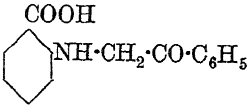
Scholtz, *Ber.*, 1918, **51**, 1652.

Phenacyl-*p*-aminobenzoic Acid.
Needles from EtOH. M.p. 211°.
Phenacyl ester: $C_{23}H_{19}O_4N$. MW, 373. M.p. 186°.
N-*Acetyl*: m.p. 176°.

Scholtz, *Ber.*, 1918, **51**, 1653.

Phenacylaniline.
See ω-Anilinoacetophenone.

Phenacylanthranilic Acid (*Phenacyl-*o-*aminobenzoic acid*)

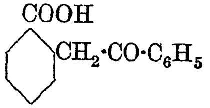


$C_{15}H_{13}O_3N$ MW, 255

Yellow needles from EtOH.Aq. M.p. 190°. Sol. ord. org. solvents.
Phenacyl ester: $C_{23}H_{19}O_4N$. MW, 373. M.p. 180°.
Phenylhydrazone: m.p. 156°.

Scholtz, *Ber.*, 1918, **51**, 1648.

***o*-Phenacylbenzoic Acid** (*Deoxybenzoin-2-carboxylic acid, phenyl* o-*carboxybenzyl ketone*)



$C_{15}H_{12}O_3$ MW, 240

Needles from EtOH.Aq. M.p. 169–70° (162–3°).
Methylamide: $C_{16}H_{15}O_2N$. MW, 253. M.p. 143–4°.

Auwers, Auffenberg, *Ber.*, 1919, **52**, 109.
Graebe, Trümpy, *Ber.*, 1898, **31**, 377.

Phenacyl bromide (*ω-Bromoacetophenone, bromomethyl phenyl ketone*)

$C_6H_5{\cdot}CO{\cdot}CH_2Br$

C_8H_7OBr MW, 199

Prisms from EtOH.Aq. M.p. 51° (50°). B.p. 135°/18 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. D^{15} 1·709. $KMnO_4 \longrightarrow$ benzoic acid.
Oxime: (i) m.p. 89·5°. *Acetyl deriv.*: m.p. 145°. (ii) M.p. 96·5–97°.
Semicarbazone: m.p. 146°.

Clibbens, Nierenstein, *J. Chem. Soc.*, 1915, **107**, 1492.
Rather, Reed, *J. Am. Chem. Soc.*, 1919, **41**, 77.
Cebrián, *Chem. Abstracts*, 1948, **42**, 8176.

Phenacylcarbinol.
See γ-Hydroxypropiophenone.

Phenacyl chloride (*ω-Chloroacetophenone, chloromethyl phenyl ketone*)

$C_6H_5 \cdot CO \cdot CH_2Cl$

C_8H_7OCl MW, 154·5

Leaflets from pet. ether. M.p. 54°. B.p. 244–5°, 139–41°/14 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. D^{15} 1·324. $CrO_3 \longrightarrow$ benzoic acid.

Oxime: m.p. 88·5–89°. *Acetyl deriv.*: m.p. 67–9°.

Semicarbazone: m.p. 156° (149°, 137°).

Hydrazone: m.p. 120°.

Clibbens, Nierenstein, *J. Chem. Soc.*, 1915, **107**, 1492.
Béhal, Detoeuf, *Compt. rend.*, 1911, **153**, 1231.

ω-Phenacylcresol.

See γ-Hydroxyphenylpropiophenone.

Phenacylformaldehyde.

See Benzoylacetaldehyde.

Phenacylglycollic Acid (*2-Benzoyl-lactic acid, 1-hydroxy-3-keto-3-phenyl-*n*-butyric acid*)

$C_6H_5 \cdot CO \cdot CH_2 \cdot CH(OH) \cdot COOH$

$C_{10}H_{10}O_4$ MW, 194

Cryst. M.p. 127° (125–6°). Sol. hot H_2O. Insol. ligroin. Hot alkalis $\longrightarrow$ oxalic acid + acetophenone.

Kozniewski, Marchlewski, *Chem. Zentr.*, 1906, II, 1190.
Koenigs, Wagstaffe, *Ber.*, 1893, **26**, 557.

Phenacylglyoxylic Acid.

See Benzoylpyruvic Acid.

Phenacylhydrazine (*ω-Hydrazinoacetophenone*)

$C_6H_5 \cdot CO \cdot CH_2 \cdot NH \cdot NH_2$

$C_8H_{10}ON_2$ MW, 150

Leaflets from C_6H_6. M.p. 85–6° decomp. Unstable at ord. temps. Sol. hot C_6H_6. Spar. sol. EtOH, Et_2O, $CHCl_3$.

B,(COOH)$_2$,H$_2$O: m.p. 149–50° decomp.

N : N′-*Diacetyl*: m.p. 123°.

ω-N-o-*Nitrobenzylidene*: m.p. 156° decomp.

ω-N-m-*Nitrobenzylidene*: m.p. 146–7°.

ω-N-o-*Hydroxybenzylidene*: m.p. 110° decomp.

Busch, Foerst, *J. prakt. Chem.*, 1928, **119**, 287.

Phenacylhydrocinnamic Acid.

See 2-Phenyl-3-benzoylbutyric Acid *and* 2-Phenyl-2′-benzoylisobutyric Acid.

Phenacylidene bromide (*ω-Dibromoacetophenone*)

$C_6H_5 \cdot CO \cdot CHBr_2$

$C_8H_6OBr_2$ MW, 278

Plates from $CHCl_3$. M.p. 35–6° (36–7°). B.p. 175–6°/23 mm. $KMnO_4 \longrightarrow$ benzoic acid.

Evans, Brooks, *J. Am. Chem. Soc.*, 1908, **30**, 406.
Wittorf, *Chem. Zentr.*, 1900, II, 29.

Phenacylidene chloride (*ω-Dichloroacetophenone*)

$C_6H_5 \cdot CO \cdot CHCl_2$

$C_8H_6OCl_2$ MW, 189

M.p. 20–21·5°. B.p. 249°, 143°/25 mm., 131–2°/11 mm. $KMnO_4 \longrightarrow$ benzoic acid.

Wittorf, *Chem. Zentr.*, 1900, II, 29.
Jackson, *J. Am. Chem. Soc.*, 1934, **56**, 977.
Gautier, *Ann. chim.*, 1888, **14**, 345, 385.
Aston, Newkirk, Jenkins, Dorsky, *Organic Syntheses*, 1943, XXIII, 48.

Phenacylidene iodide (*ω-Di-iodoacetophenone*)

$C_6H_5 \cdot CO \cdot CHI_2$

$C_8H_6OI_2$ MW, 372

Oil. B.p. above 200° decomp. Sol. EtOH, Et_2O, C_6H_6, CS_2. Insol. H_2O.

Collet, *Bull. soc. chim.*, 1900, **23**, 830.
Wolff, *Ann.*, 1902, **325**, 143.

2-Phenacylidenemethylfuran.

See Furfurylideneacetophenone.

Phenacyl iodide (*ω-Iodoacetophenone, iodomethyl phenyl ketone*)

$C_6H_5 \cdot CO \cdot CH_2I$

C_8H_7OI MW, 246

M.p. 34°. B.p. 158–60°/15 mm.

Truchet, *Ann. chim.*, 1931, **16**, 373.
Rheinboldt, Perrier, *J. Am. Chem. Soc.*, 1947, **69**, 3148.

Phenacyl Mercaptan (*ω-Mercaptoacetophenone*)

$C_6H_5 \cdot CO \cdot CH_2SH$

C_8H_8OS MW, 152

M.p. 23–4°. B.p. 116–22°/4 mm. D_4^{20} 1·1753. Readily decomp. $\longrightarrow H_2S$. ZnHg + HCl $\longrightarrow$ ethylbenzene quantitatively.

Oxime: m.p. 70°.

Phenylhydrazone: m.p. 90–1°.

Groth, *Chem. Abstracts*, 1924, **18**, 1281.

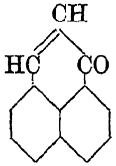
Phenacylphenol.

See Hydroxydeoxybenzoin.

1-Phenacylpropionic Acid.

See 2-Benzoylisobutyric Acid.

Phenalone (peri-*Naphthindenone*, peri-*naphthindone*)



$C_{13}H_8O$ MW, 180

Canary-yellow leaflets from MeOH or cyclohexene. M.p. 153–4°. Basic. Sol. conc. HCl, + $H_2O \longrightarrow$ red col. Conc. $H_2SO_4 \longrightarrow$ yellow sol. with intense green fluor.

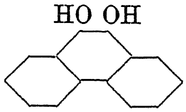
Semicarbazone: m.p. 210° decomp.

Hydrazone: dark brownish-yellow needles from EtOH. M.p. 125–30° decomp.

Badische, D.R.P., 283,066, (*Chem. Zentr.*, 1915, I, 814).
Cook, Hewitt, *J. Chem. Soc.*, 1934, 368, 373.
Lulsin, *Chem. Abstracts*, 1942, **36**, 454.
See also Braun, Manz, Reinsch, *Ann.*, 1929, **468**, 301.

Phenanthracridine.
See 1 : 2 : 3 : 4-Dibenzacridine.

Phenanthrahydroquinone (9 : 10-*Dihydroxyphenanthrene, phenanthraquinol, phenanthrene*-9 : 10-*diol*)



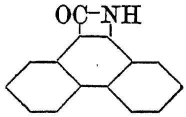
$C_{14}H_{10}O_2$ MW, 210

Needles. M.p. 148°. Sol. EtOH, Et_2O, C_6H_6, hot H_2O. Heat of comb. C_v 1604·3 Cal. Ox. in air ⟶ 9 : 10-phenanthraquinone.
Monoacetyl: m.p. 168–70°.
Diacetyl: m.p. 202°.
Dibenzoyl: m.p. 216–17°.
Mono-o-*xylyl ether*: $C_{22}H_{18}O_2$. MW, 314. Yellow cryst. from ligroin. M.p. 148–9°.
Mono-p-*xylyl ether*: yellow needles from ligroin. M.p. 129–30°.

Braun, Bayer, *Ber.*, 1925, **58**, 2680.
Benrath, Meyer, *J. prakt. Chem.*, 1914, **89**, 258.
Schmidt, Lumpp, *Ber.*, 1910, **43**, 790.

Phenanthrane.
See 9 : 10-Dihydrophenanthrene.

Phenanthranil (*Phenanthranilic acid lactam*)

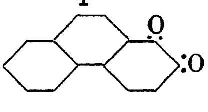


$C_{15}H_9ON$ MW, 219

Needles from C_6H_6. M.p. 241°. Sol. C_6H_6. Sublimes. Na_2CO_3 ⟶ Na salt of acid. Fuming HCl at 170° ⟶ 9-hydroxyphenanthrene.
N-*Acetyl*: m.p. 145°.
Et ether: $C_{17}H_{13}ON$. MW, 247. Needles from pet. ether. M.p. 110°. Sol. EtOH, C_6H_6.

Japp, Knox, *J. Chem. Soc.*, 1905, **87**, 692.

1 : 2-Phenanthraquinone

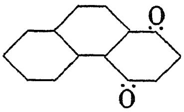


$C_{14}H_8O_2$ MW, 208

Needles from toluene. M.p. 222° decomp. Sol. AcOH. Mod. sol. EtOH, C_6H_6. H_2SO_4 ⟶ blue col. changing rapidly to green.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 1896.

1 : 4-Phenanthraquinone

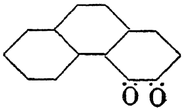


$C_{14}H_8O_2$ MW, 208

Yellow needles from ligroin. M.p. 155°. Sol. EtOH, C_6H_6, AcOH. H_2SO_4 ⟶ violet-red col.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2469.

3 : 4-Phenanthraquinone

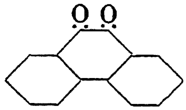


$C_{14}H_8O_2$ MW, 208

Red needles from C_6H_6–ligroin. M.p. 133° decomp. H_2SO_4 ⟶ Prussian-blue col. changing to chrome-green. Red. ⟶ morphol.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 946.

9 : 10-Phenanthraquinone

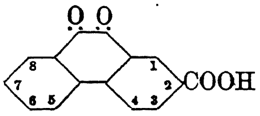


$C_{14}H_8O_2$ MW, 208

Orange-yellow needles. M.p. 206–207·5° (205°, 202°). B.p. above 360°. Sol. Et_2O, hot AcOH. Spar. sol. EtOH, C_6H_6, AcOEt. Insol. H_2O. Sol. conc. H_2SO_4 with dull green col. Sublimes in orange-red plates. $NaHSO_3$ gives unstable bisulphite comp. Ox. ⟶ diphenic acid. Red. ⟶ phenanthrahydroquinone.
Monoxime: m.p. 158°.
Dioxime: m.p. 202° decomp. (rapid heat). *Me ether*: m.p. 222–3°. *Di-Me ether*: m.p. 145–6°. *Diacetyl deriv.*: m.p. 184°.
Monosemicarbazone: m.p. about 220° decomp.
Mono-p-*nitrophenylhydrazone*: m.p. 245°.

Lewis, Gibbs, U.S.P., 1,288,431, (*Chem. Abstracts*, 1919, **13**, 451).
Tseng, Chu, *Chem. Abstracts*, 1934, **28**, 3730.
Selden, B.P. 170,022, (*Chem. Abstracts*, 1922, **16**, 1137).
Scholl, Schwarzer, *Ber.*, 1922, **55**, 324.
Auwers, *Ann.*, 1911, **378**, 210.
Kenner, Wilson, *J. Chem. Soc.*, 1927, 1111.

Phenanthraquinone-2-carboxylic Acid



$C_{15}H_8O_4$ MW, 252

Red needles from AcOH. Does not melt below 310°. Spar. sol. hot EtOH, hot Me_2CO. Insol. Et_2O, C_6H_6, CS_2.

Nitrile: $C_{15}H_7O_2N$. MW, 233. Reddish-yellow leaflets from AcOH. M.p. 290°.

Liebermann, Kardos, *Ber.*, 1913, **46**, 201.
Werner, *Ann.*, 1902, **321**, 356.

Phenanthraquinone-3-carboxylic Acid.

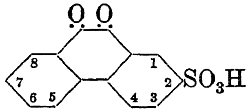
Reddish-yellow cryst. from hot AcOH. Does not melt below 315°.

Amide: $C_{15}H_9O_3N$. MW, 251. Orange-red needles. M.p. 290°.

Nitrile: $C_{15}H_7O_2N$. MW, 233. Orange cryst. from AcOH. M.p. 282–3°.

Werner, *Ann.*, 1902, **321**, 355.

Phenanthraquinone-2-sulphonic Acid



$C_{14}H_8O_5S$ MW, 288

Me ester: $C_{15}H_{10}O_5S$. MW, 302. Yellow leaflets from AcOH. M.p. 196–7° (192–192·5°).

Chloride: $C_{14}H_7O_4ClS$. MW, 306·5. Yellow leaflets or needles from AcOH. M.p. 245–6° decomp.

Sandqvist, *Ann.*, 1911, **379**, 89.

Phenanthraquinone-3-sulphonic Acid.

Me ester: orange-yellow cryst. from AcOH. M.p. 235°.

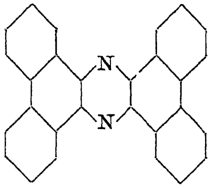
Chloride: yellowish-red needles. M.p. 257° (228–32° decomp.).

Sandqvist, *Ann.*, 1913, **398**, 136.
Werner, *Ann.*, 1902, **321**, 341.

Phenanthraquinol.

See Phenanthrahydroquinone.

Phenanthrazine

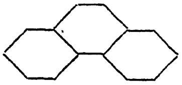


$C_{28}H_{16}N_2$ MW, 380

Yellow cryst. from tetralin. M.p. 487°. Conc. H_2SO_4 ⟶ deep cornflower blue col.

Schiedt, *J. prakt. Chem.*, 1941, **157**, 203.

Phenanthrene



$C_{14}H_{10}$ MW, 178

Constituent of crude anthracene fraction of coal tar. Plates from EtOH. M.p. 101° (98·7–99·5°, 96·5–97·5°). B.p. 340° (332), 210–15°/12 mm. Sol. Et_2O, C_6H_6, $CHCl_3$, Me_2CO, CS_2, CCl_4. Mod. sol. EtOH, MeOH, AcOH, pet. ether. Insol. H_2O. Sols. show blue fluor. D^4 0·9800. n_D 1·59427. Sublimes in leaflets. Ox. ⟶ 9 : 10-phenanthraquinone ⟶ diphenic acid.

Picrate: m.p. 144° (132·8°).

$C_{14}H_{10} + C_6H_2Cl(NO_2)_3$-2 : 1 : 3 : 5 : m.p. 82·4°.

$C_{14}H_{10},C_6H_3(NO_2)_3$-1 : 3 : 5 : m.p. 158°

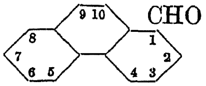
$C_{14}H_{10},C_6H_2(NO_2)_3NH_2$: m.p. 160–2°.

Zelinsky, Titz, Gaverdovshaia, *Ber.*, 1926, **59**, 2590.
Clar, *Ber.*, 1932, **65**, 846, 1411.
Hönig, *Z. Elektrochem.*, 1929, **35**, 847.
Ruzicka, *Helv. Chim. Acta*, 1934, **17**, 473.
Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 557.
Nikolaev, *Chem. Abstracts*, 1935, **29**, 7972.
Fieser, Hershberg, *J. Am. Chem. Soc.*, 1935, **57**, 1508.
Iljinski, *Chem. Zentr.*, 1932, I, 1191.

Phenanthrene-acetic Acid.

See Phenanthrylacetic Acid.

Phenanthrene-1-aldehyde



$C_{15}H_{10}O$ MW, 206

M.p. 110–11°.

Oxime: m.p. 187–9°.

Bachmann, Kloetzel, *J. Am. Chem. Soc.*, 1937, **59**, 2207.

Phenanthrene-2-aldehyde.

Needles. M.p. 59–59·5°. Sol. EtOH, MeOH.

Oxime: m.p. 194–5°.

Semicarbazone: m.p. 281–2°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996.

Phenanthrene-3-aldehyde.

Leaflets from C_6H_6–pet. ether. M.p. 79·5–80°. Sol. EtOH, MeOH.

Oxime: m.p. 145–145·5°.

Semicarbazone: m.p. 274–5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996.

Phenanthrene-9-aldehyde.

Yellow prisms from EtOH. M.p. 100–1°. B.p. 231–3°/12 mm. Mod. sol. EtOH.

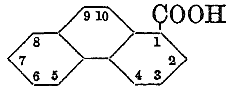
Oxime: m.p. 157–157·5° (156–7°).

Semicarbazone: m.p. 221–2° (222–222·5°).

p-*Nitrophenylhydrazone*: m.p. 265°.

Hinkel, Ayling, Beynon, *J. Chem. Soc.*, 1936, 344.
Miller, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 769.

Phenanthrene-1-carboxylic Acid (1-*Phenanthroic acid*)



$C_{15}H_{10}O_2$ MW, 222

Needles from EtOH. M.p. 232–3°. Sol. C_6H_6.

Me ester: $C_{16}H_{12}O_2$. MW, 236. M.p. 57°. Sol. ord. org. solvents.

Amide: $C_{15}H_{11}ON$. MW, 221. Plates from AcOH.Aq. M.p. 284°.

Nitrile: $C_{15}H_9N$. MW, 203. Needles from EtOH. M.p. 128°.

Anilide: m.p. 245°.

Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1381.
Fieser, *J. Am. Chem. Soc.*, 1932, **54**, 4110.

Phenanthrene-2-carboxylic Acid (2-*Phenanthroic acid*).

Needles from AcOH. M.p. 258·5–260°. Sol. EtOH, AcOH.

Me ester: needles from MeOH. M.p. 96–96·5°.

Et ester: $C_{17}H_{14}O_2$. MW, 250. Needles from pet. ether. M.p. 73–73·5°.

Chloride: $C_{15}H_9OCl$. MW, 240·5. M.p. 101–101·5°.

Amide: needles from C_6H_6. M.p. 242–3°.

Nitrile: cryst. from EtOH. M.p. 108–109·5°.

Anilide: m.p. 217–18°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996; 1930, **52**, 3708.
Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 558.

Phenanthrene-3-carboxylic Acid (3-*Phenanthroic acid, α-phenanthrene-carboxylic acid*).

Needles from AcOH. M.p. 269° (270°). Sol. EtOH, Et_2O, AcOH. Insol. H_2O. Sublimes.

Me ester: needles from MeOH. M.p. 94·5–95° (97°).

Et ester: needles from pet. ether. M.p. 56–7°.

Chloride: m.p. 116–17°.

Amide: cryst. from EtOH. M.p. 233–4° (227–8°).

Nitrile: needles from EtOH. M.p. 102°.

Anilide: needles from Me_2CO. M.p. 216–17°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1930, **52**, 3710; 1933, **55**, 2996.
Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 558.
Kruber, *Ber.*, 1934, **67**, 1005.

Phenanthrene-9-carboxylic Acid (9-*Phenanthroic acid, β-phenanthrene-carboxylic acid*).

Cryst. from EtOH. M.p. 252°. Sol. EtOH, Et_2O, AcOH. Insol. H_2O. Sublimes.

Me ester: m.p. 116°.

Et ester: m.p. 61°.

Chloride: m.p. 102°. B.p. 240°/13 mm.

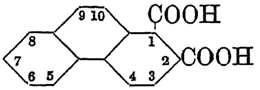
Amide: needles from C_6H_6. M.p. 232–3° (226°).

Nitrile: needles from EtOH. M.p. 103°.

Anilide: m.p. 218°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996.
Shoppee, *J. Chem. Soc.*, 1933, 39.
Bachmann, *J. Am. Chem. Soc.*, 1934, **56**, 1366.

Phenanthrene-1 : 2-dicarboxylic Acid



$C_{16}H_{10}O_4$ MW, 266

Di-Me ester: yellow cryst. from MeOH. M.p. 132·5–133°.

Di-Et ester: pale yellow cryst. from MeOH. M.p. 132°.

Anhydride: $C_{16}H_8O_3$. MW, 248. Yellow needles from Ac_2O. M.p. 311–13°.

Fieser, Hershberg, *J. Am. Chem. Soc.*, 1935, **57**, 1508, 1853.

Phenanthrene-1 : 7-dicarboxylic Acid.

Di-Me ester: $C_{18}H_{14}O_4$. MW, 294. Cryst. from MeOH. M.p. 151–2°.

Ruzicka, Graaff, Hosking, *Helv. Chim. Acta*, 1931, **14**, 238.

Phenanthrene-3 : 4-dicarboxylic Acid.

Di-Me ester: prisms from Et_2O–pet. ether. M.p. 114·5–114·8°.

Anhydride: lemon-yellow needles from xylene. M.p. 253·5–254°.

Fieser, Fieser, Hershberg, *J. Am. Chem. Soc.*, 1936, **58**, 2322.

Phenanthrene-3 : 9-dicarboxylic Acid.

Di-Me ester: needles from MeOH. M.p. 126–7°.

Schultz *et al.*, *J. Org. Chem.*, 1946, **11**, 307.

Phenanthrene-9 : 10-dicarboxylic Acid.

Di-Me ester: pale yellow plates from EtOH. M.p. 131°.

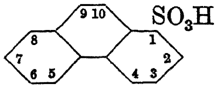
Anhydride: yellow rods from Ac_2O. M.p. 322°.

Jeanes, Adams, *J. Am. Chem. Soc.*, 1937, **59**, 2608.

Phenanthrene-propionic Acid.

See Phenanthrylpropionic Acid.

Phenanthrene-1-sulphonic Acid



$C_{14}H_{10}O_3S$ MW, 258

p-*Toluidine salt*: needles. M.p. 267°.

Me ester: $C_{15}H_{12}O_3S$. MW, 272. Plates from MeOH. M.p. 102°.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

Phenanthrene-2-sulphonic Acid.

Cryst. + $1H_2O$. M.p. about 150°. Sol. hot toluene, hot $PhNO_2$.

p-*Toluidine salt*: m.p. 291°.

Me ester: (i) m.p. 101·5°. (ii) Leaflets. M.p. 96–8°. Sol. MeOH, EtOH.

Et ester: $C_{16}H_{14}O_3S$. MW, 286. Yellowish-brown leaflets from EtOH. M.p. 88·5°.

Chloride: $C_{14}H_9O_2ClS$. MW, 276·5. Leaflets from AcOH. M.p. 156°.

Amide: $C_{14}H_{11}O_2NS$. MW, 257. Leaflets from EtOH. M.p. 253–4°.

Anilide: m.p. 157–8°.

Sandqvist, *Ann.*, 1911, **379**, 79.
Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.
Ioffe, *Chem. Abstracts*, 1934, **28**, 1694.

Phenanthrene-3-sulphonic Acid (α-*Phenanthrenesulphonic acid*).

Cryst. from H_2O ⟶ two hydrates. (i) + $1H_2O$, m.p. 120–1°: (ii) + $2H_2O$, m.p. 88–9°. M.p. anhyd. 175–6°.

p-*Toluidine salt*: m.p. 222°.

Me ester: cryst. from EtOH. M.p. 119–20°.

Et ester: leaflets or needles. M.p. 107–8°. Sol. EtOH, C_6H_6.

Chloride: yellow leaflets from C_6H_6. M.p. 110–11° (108·5°): solidifies, and remelts at 114°.

Bromide: $C_{14}H_9O_2BrS$. MW, 321. Yellow plates from ligroin. M.p. 140°.

Amide: leaflets from EtOH.Aq. M.p. 189·5–190°.

Sandqvist, *Ann.*, 1909, **369**, 104; 1913, **398**, 136.
Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

Phenanthrene-9-sulphonic Acid (β-*Phenanthrenesulphonic acid*).

Leaflets or needles + $2H_2O$ from H_2O or C_6H_6. M.p. 134°, 174° anhyd.

p-*Toluidine salt*: needles. M.p. 235°.

Me ester: leaflets from MeOH. M.p. 106°.

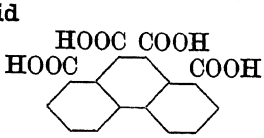
Et ester: cryst. from EtOH. M.p. 108°.

Chloride: yellow needles from AcOH. M.p. 127° (125·5°).

Amide: needles from EtOH. M.p. 193·5°.

Sandqvist, *Ann.*, 1912, **392**, 76.
Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.
Ioffe, *Chem. Abstracts*, 1934, **28**, 1694.

Phenanthrene-1 : 8 : 9 : 10-tetracarboxylic Acid



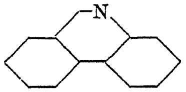
$C_{18}H_{10}O_8$ MW, 354

Tetra-Me ester: $C_{22}H_{18}O_8$. MW, 410. Yellow cryst. M.p. 181–3°.

Di-anhydride: $C_{18}H_6O_6$. MW, 318. Brownish-yellow needles. Does not melt below 400°.

Zinke, Hauswirth, Grimm, *Monatsh.*, 1931, **57**, 405.

Phenanthridine (3 : 4-*Benzquinoline*, 2 : 3 : 4 : 5-*dibenzpyridine*, 9-*azaphenanthrene*)



$C_{13}H_9N$ MW, 179

Needles from EtOH.Aq. M.p. 106°. B.p. 349°/769 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, CS_2. Spar. sol. H_2O with blue fluor. Spar. volatile in steam.

$B,HCl,HgCl_2$: m.p. 197°.

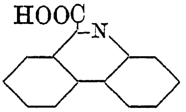
B_2,H_2PtCl_6: m.p. above 225°.

Methiodide: m.p. 204·5°.

Picrate: m.p. 220°.

Sielisch, Sandke, *Ber.*, 1933, **66**, 433.
Pyl, *Ber.*, 1927, **60**, 287.
Ritchie, *J. Proc. Roy. Soc. N.S. Wales*, 1945, **78**, 164, (*Chem. Abstracts*, 1946, **40**, 880).

Phenanthridine-9-carboxylic Acid



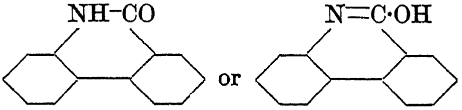
$C_{14}H_9O_2N$ MW, 223

Micro-cryst. Sol. EtOH, Me_2CO. Spar. sol. C_6H_6. Insol. H_2O, pet. ether. Heat ⟶ phenanthridine.

Et ester: $C_{16}H_{13}O_2N$. MW, 251. Needles from pet. ether. M.p. 57–8°.

Walls, *J. Chem. Soc.*, 1934, 108.

Phenanthridone (*Phenanthridol*, 10-*hydroxyphenanthridine*)

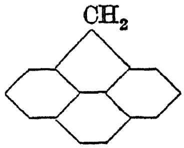


$C_{13}H_9ON$ MW, 195

Needles from EtOH. M.p. 293°. Spar. sol. EtOH, Et_2O, AcOH. Zn dist. ⟶ phenanthridine.

Oyster, Adkins, *J. Am. Chem. Soc.*, 1921, **43**, 210.
Meyer, Hofmann, *Monatsh.*, 1916, **37**, 701.
Walls, *J. Chem. Soc.*, 1934, 108; 1935, 1407.
Heidenreich, Tust, U.S.P., 1,880,441, (*Chem. Abstracts*, 1933, **27**, 516).
See also Sielich, Sandke, *Ber.*, 1933, **66**, 434.

Phenanthrindene (*Phenanthrylenemethane*)



$C_{15}H_{10}$ MW, 190

Needles from EtOH. M.p. 116°. B.p. 353°.
Benzylidene deriv.: m.p. 108°.
Picrate: m.p. 166°.

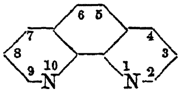
Kruber, *Ber.*, 1934, **67**, 1004.

Phenanthroic Acid.
See Phenanthrene-carboxylic Acid.

Phenanthrol.
See Hydroxyphenanthrene.

Phenanthrol-carboxylic Acid.
See Hydroxyphenanthrene-carboxylic Acid.

o-Phenanthroline (1 : 10-*Phenanthroline*)

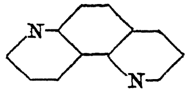


$C_{12}H_8N_2$ MW, 180

Cryst. + $1H_2O$ from H_2O, m.p. 91·5°; cryst. from C_6H_6, m.p. 98–100°. B.p. above 300°. Sol. EtOH, C_6H_6, Me_2CO. Insol. pet. ether. Ferrous salts ⟶ red col. Forms metallic derivs.

Tartarini, Samaja, *Chem. Abstracts*, 1933, **27**, 5741.
Smith, Getz, *Chem. Reviews*, 1935, **16**, 113.
Willink, Wibaut, *Rec. trav. chim.*, 1935, **54**, 282.
Smith, *J. Am. Chem. Soc.*, 1930, **52**, 402.

***m*-Phenanthroline** (1 : 7-*Phenanthroline*)

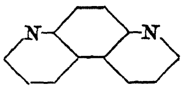


$C_{12}H_8N_2$ MW, 180

Needles. M.p. 65·5°, anhyd. 78–78·5°. B.p. above 360°. Sol. EtOH, hot H_2O. Insol. Et_2O, C_6H_6, ligroin.

Knüppel, *Ber.*, 1896, **29**, 707.
See also Smith, *J. Am. Chem. Soc.*, 1930, **52**, 397.

***p*-Phenanthroline** (*ψ-Phenanthroline*, 4 : 7-*phenanthroline*)



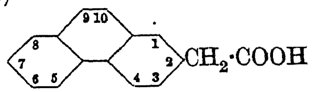
$C_{12}H_8N_2$ MW, 180

Needles from H_2O. M.p. anhyd. 173°. Sol. EtOH, $CHCl_3$. Mod. sol. hot H_2O. Spar. sol. Et_2O, C_6H_6, CS_2.
Hydrochloride: does not melt below 315°.
Sulphate: m.p. 233–4°.
Chromate: decomp. at 225–30°.
Picrate: m.p. 255–6°.
Platinochloride: does not melt below 310°.
Methiodide: m.p. 268–9°.
Di-methiodide: m.p. 271°.

Matsumura, *J. Am. Chem. Soc.*, 1930, 52, 3196.
Smith, *ibid.*, 402.
Kabachnik, Reson, *J. Applied Chem., U.S.S.R.*, 1936, **9**, 2026, (*Chem. Abstracts*, 1937, **31**, 2608).

Phenanthrone.
Keto form of 9-Hydroxyphenanthrene, *q.v.*

2-Phenanthrylacetic Acid (*Phenanthrene*-2-*acetic acid*)



$C_{16}H_{12}O_2$ MW, 236

Needles. M.p. 183·5–184·5°.
Me ester: $C_{17}H_{14}O_2$. MW, 250. Needles. M.p. 78–78·5°.
Nitrile: $C_{16}H_{11}N$. MW, 217. M.p. 106–106·5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2998.

3-Phenanthrylacetic Acid (*Phenanthrene*-3-*acetic acid*).
Leaflets. M.p. 177–177·5°.
Me ester: liq. *Picrate*: m.p. 103·5–104°.
Nitrile: m.p. 84·5–85°.

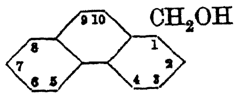
Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2998.

9-Phenanthrylacetic Acid (*Phenanthrene*-9-*acetic acid*).
Leaflets. M.p. 220–1° (213–15°).
Me ester: m.p. 75–75·5°.
Amide: $C_{16}H_{13}ON$. MW, 235. Leaflets. M.p. 250–2°.
Nitrile: m.p. 96·5–97°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2998.
Willgerodt, Albert, *J. prakt. Chem.*, 1911, **84**, 387

Phenanthrylamine.
See Aminophenanthrene.

1-Phenanthrylcarbinol (1-*Hydroxymethylphenanthrene*)



$C_{15}H_{12}O$ MW, 208

M.p. 165°.

Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1382.

2-Phenanthrylcarbinol (*2-Hydroxymethyl-phenanthrene*).

Needles from C_6H_6–pet. ether. M.p. 125–125·5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2998.

3-Phenanthrylcarbinol (*3-Hydroxymethyl-phenanthrene*).

Prisms from C_6H_6–pet. ether. M.p. 103–103·5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2998.

9-Phenanthrylcarbinol (*9-Hydroxymethyl-phenanthrene*).

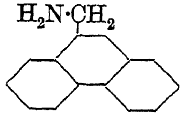
Needles from C_6H_6–pet. ether. M.p. 149–149·5°.

Bachmann, *J. Am. Chem. Soc.*, 1934, **56**, 1366.

Phenanthrylene-methane.

See Phenanthrindene.

9-Phenanthrylmethylamine (*9-Amino-methylphenanthrene*)

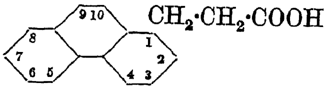


$C_{15}H_{13}N$ MW, 207

Cryst. from Et_2O–ligroin. M.p. 107°.
N-*Acetyl*: m.p. 182–5°.
N-*Benzoyl*: m.p. 167°.
N-*Benzylidene*: m.p. 103·5°.
Picrate: m.p. 241° decomp.

Shoppee, *J. Chem. Soc.*, 1933, 40.

β-1-Phenanthrylpropionic Acid (*Phenan-threne-1-propionic acid*)



$C_{17}H_{14}O_2$ MW, 250

Leaflets from AcOH.Aq. M.p. 188·5–189°.
Me ester: plates from AcOMe–MeOH. M.p. 90–1°.

Bachmann, Kloetzel, *J. Am. Chem. Soc.*, 1937, **59**, 2207.
Natelson, Gottfried, *ibid.*, 216.

β-2-Phenanthrylpropionic Acid (*Phenan-threne-2-propionic acid*).

Leaflets from AcOH.Aq. M.p. 177–177·5°.
Me ester: plates from AcOMe–MeOH. M.p. 82–3°.

Bachmann, Kloetzel, *J. Am. Chem. Soc.*, 1937, **59**, 2207.

β-3-Phenanthrylpropionic Acid (*Phenan-threne-3-propionic acid*).

Plates from C_6H_6. M.p. 158·5–159·5°.
Me ester: plates from MeOH. M.p. 63–4°.

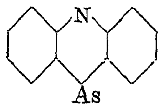
Bachmann, Kloetzel, *J. Am. Chem. Soc.*, 1937, **59**, 2207.

β-9-Phenanthrylpropionic Acid (*Phenan-threne-9-propionic acid*).

Needles from C_6H_6. M.p. 173–4°.
Me ester: needles from MeOH. M.p. 72–3°.

Bachmann, Kloetzel, *J. Am. Chem. Soc.*, 1937, **59**, 2207.

Phenarsazine (*Phenazarsine*)



$C_{12}H_8NAs$ MW, 241

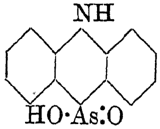
M.p. about 310°. Sol. $PhNO_2$. Spar. sol. hot xylene.

Wieland, Rheinheimer, *Ann.*, 1921, **423**, 16.

"Phenarsazine chloride."

See 10-Chloro-5 : 10-dihydrophenarsazine.

Phenarsazinic Acid (*Phenazarsinic acid*)

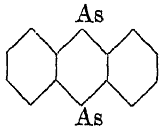


$C_{12}H_{10}O_2NAs$ MW, 275

Cryst. from H_2O. M.p. 365–6° decomp.
B,HCl: m.p. 208°.

Kappelmeier, *Rec. trav. chim.*, 1930, **49**, 83 (*Bibl.*).
Sergeev, Gorkii, *Chem. Abstracts*, 1932, **26**, 2195.
Wieland, Rheinheimer, *Ann.*, 1921, **423**, 22.
See also Razuvaev, Koton, *Chem. Abstracts*, 1933, **27**, 984.

Phenarsine (*Arsanthrene, diarseno-9 : 10-anthracene*)



$C_{12}H_8As_2$ MW, 302

Orange-yellow leaflets. M.p. about 340°. Spar. sol. AcOH, Py.

Kalb, *Ann.*, 1921, **423**, 66.

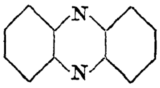
Phenazarsine.

See Phenarsazine.

Phenazarsinic Acid.

See Phenarsazinic Acid.

Phenazine (*Azophenylene*)



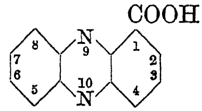
$C_{12}H_8N_2$ MW, 180

Yellowish-red needles from AcOH. M.p. 171° (170°). B.p. above 360°. Sol. hot EtOH. Mod. sol. Et_2O, C_6H_6. Spar. sol. H_2O. Sublimes. Volatile in steam.

Di-N-*oxide* : m.p. 204° decomp.

Eckert, Steiner, *Monatsh.*, 1914, **35**, 1153.
Zerewitinoff, Ostromisslensky, *Ber.*, 1911, **44**, 2402.
Kehrmann, Mermod, *Helv. Chim. Acta*, 1927, **10**, 62.
Beschke, *Ann.*, 1913, **398**, 298.
Clemo, McIlwain, *J. Chem. Soc.*, 1934, 1993.

Phenazine-1-carboxylic Acid



$C_{13}H_8O_2N_2$ MW, 224

Yellow needles from EtOH. M.p. 239°.
Amide : $C_{13}H_9ON_3$. MW, 223. M.p. 234°.

Clemo, McIlwain, *J. Chem. Soc.*, 1934, 1993.
Kögl, Tönnis, Groenewegen, *Ann.*, 1932, **497**, 277.

Phenazine-2-carboxylic Acid.
Yellow needles from Me_2CO. M.p. 292–3°.
Amide : $C_{13}H_9ON_3$. MW, 223. M.p. 312°.

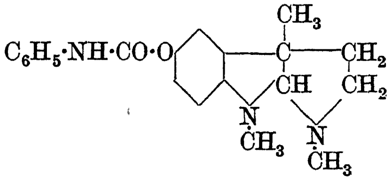
Kögl, Tönnis, Groenewegen, *Ann.*, 1932, **497**, 288.
Clemo, McIlwain, *J. Chem. Soc.*, 1935, 741.
McIlwain, *J. Chem. Soc.*, 1937, 1701.

Phenazone.
See Antipyrine.

Phenazoxine.
See Phenoxazine.

Phendioxin.
See Diphenylene dioxide.

Pheneserine (*Eseroline phenylurethane*)



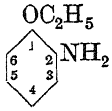
$C_{20}H_{23}O_2N_3$ MW, 337

Prisms. M.p. 150°. NaOH ⟶ eseroline.
Methiodide : m.p. 198°. $[\alpha]_D$ − 92·8° in EtOH.
Picrate : non-cryst. M.p. about 100°.

Polonovski, *Bull. soc. chim.*, 1916, **19**, 46.

Phenetetrol.
See Apionol.

***o*-Phenetidine** (o-*Ethoxyaniline*, o-*aminophenetole*, o-*aminophenol ethyl ether*)



$C_8H_{11}ON$ MW, 137

B.p. 228° (224–9°). $k = 4{\cdot}64 \times 10^{-10}$ at 20°.
B,$C_{10}H_7SO_3H$ (1) : m.p. 185°.
B,$C_{10}H_7SO_3H$ (2) : m.p. 197°.
N-*Formyl* : form-*o*-phenetidide. M.p. 62°. B.p. 292°.
N-*Acetyl* : acet-*o*-phenetidide, *o*-ethoxyacetanilide. $C_{10}H_{13}O_2N$. MW, 179. M.p. 79°.
N-*Lauryl* : m.p. 69·7–70°.
N-*Myristyl* : m.p. 77°.
N-*Palmityl* : m.p. 82–3°.
N-*Stearyl* : m.p. 84·7°.
N-*Benzoyl* : benz-*o*-phenetidide. M.p. 104·2°.
N-1-*Naphthalenesulphonyl* : m.p. 185°.
N-2-*Naphthalenesulphonyl* : m.p. 197°.
p-*Iodophenylurea* : m.p. 175–6°.
2-*Nitro*-1 : 3-*indandione deriv.* : m.p. 206°.

Zavelskii, Fomenko, Krolik, *Chem. Abstracts*, 1935, **29**, 467.
Li, Adams, *J. Am. Chem. Soc.*, 1935, **57**, 1568.
Birosel, Huang, *Chem. Abstracts*, 1933, **27**, 5727.

***m*-Phenetidine** (m-*Ethoxyaniline*, m-*aminophenetole*, m-*aminophenol ethyl ether*).
B.p. 248°, 127–8°/11 mm.
N-*Formyl* : form-*m*-phenetidide. Greyish-blue cryst. M.p. 52°.
N-*Acetyl* : acet-*m*-phenetidide. Greyish plates. M.p. 96–7°.
N-*Benzoyl* : see under *m*-Hydroxybenzanilide.
N-*Toluene*-p-*sulphonyl* : m.p. 157°.
Picrate : m.p. 158°.
2-*Nitro*-1 : 3-*indandione deriv.* : m.p. 210°.

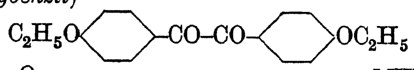
Reverdin, Lokietek, *Bull. soc. chim.*, 1915, **17**, 407.

***p*-Phenetidine** (p-*Ethoxyaniline*, p-*aminophenetole*, p-*aminophenol ethyl ether*).
M.p. 2·4°. B.p. 254·2–254·7°. $k = 2{\cdot}15 \times 10^{-9}$ at 15°. $D_4^{15\cdot9}$ 1·0652. Spar. volatile in steam.
B,HCl : m.p. 234°. Sol. H_2O. Sublimes.
B,$(COOH)_2$: m.p. 201°.
B_2,$(COOH)_2$: m.p. 201°.
Tartrate : vinopyrin. M.p. 192°.
N-*Formyl* : form-*p*-phenetidide. M.p. 68–70°.
N-*Acetyl* : acet-*p*-phenetidide. Phenacetin. $C_{10}H_{13}O_2N$. MW, 179. M.p. 137–8°. Sol. 1400 parts H_2O at 15°, 80 parts at boil. Sol. 6 parts Py at 20°, 67 parts Et_2O at 25°, 20 parts $CHCl_3$ at 25°, 10 parts Me_2CO at 30°, 170 parts C_6H_6 at 30°, 6·5 parts EtOH. Heat of comb. C_p 1303 Cal. NaOBr + NH_3 ⟶ brown col.

Forms cryst. K deriv. Antipyretic and antineuralgic. *B_2,HI* : needles. M.p. 147–8°.
N-*Lactyl* : *see under* Lactic acid.
N-*Glycyl* : phenocoll. Needles + $1H_2O$. M.p. 95°, 100·5° anhyd.
N-*Lauryl* : m.p. 109–10°.
N-*Palmityl* : m.p. 117·5°.
N-*Stearyl* : m.p. 112·5°.
Glucoside : m.p. 114°.
N-*Furfurylidene* : m.p. 72–3°.
N-*Benzoyl* : *see under* *p*-Hydroxybenzanilide.
N-*Mandelyl* : *see* Amygdophenine.
N-1-*Naphthalenesulphonyl* : m.p. 201–2°.
N-2-*Naphthalenesulphonyl* : m.p. 207°.
N-*Me* : *see* *N*-Methyl-*p*-phenetidine.
p-*Iodophenylurea* : m.p. 260°.
2-*Nitro*-1 : 3-*indandione deriv.* : m.p. 190°.

Täufel, Wagner, Dünwald, *Z. Elektrochem.*, 1928, **34**, 115.
Grether, U.S.P., 1,722,417, (*Chem. Abstracts*, 1929, **23**, 4483).
Birosel, Huang, *Chem. Abstracts*, 1933, **27**, 5727.
Ehrhardt, *Ber.*, 1897, **30**, 2015.
Byk, D.R.P., 264,263, (*Chem. Zentr.*, 1913, II, 1179).
Zernik, *Chem. Zentr.*, 1908, I, 1203.
Aoyama, Eguchi, Tashiro, *Chem. Abstracts*, 1935, **29**, 5427.
Zavelskii, Fomenko, Krolik, *ibid.*, 467.
West, *J. Chem. Soc.*, 1925, 494.
Hinsberg, *Ann.*, 1899, **305**, 278.
Tauber, D.R.P., 85,988.
Lukashevich, Voroshilova, *Org. Chem. Ind. U.S.S.R.*, 1937, **4**, 253, (*Chem. Abstracts*, 1938, **32**, 2091).

p-Phenetil (*Di-*[*4-ethoxy*]*-benzoyl, 4 : 4′-diethoxybenzil*)



$C_{18}H_{18}O_4$ MW, 298

Prisms from EtOH. M.p. 149°.
Monoxime : m.p. 136°.
Di-phenylhydrazone : m.p. 171°.

Vorländer, *Ber.*, 1911, **44**, 2464.

Phenetole (*Ethyl phenyl ether, ethoxybenzene*)

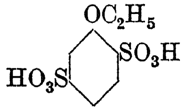
$C_6H_5{\cdot}OC_2H_5$

$C_8H_{10}O$ MW, 122

F.p. $-$ 28·6° ($-$ 30·2°). M.p. $-$ 33°. B.p. 172°, 166°/667·5 mm., 160°/565·5 mm., 92·5°/61·42 mm., 77·5°/31·14 mm., 60°/9·12 mm. Sol. EtOH, Et_2O. Insol. H_2O. Heat of comb. C_p 1057·23 Cal. $D_4^{20\cdot2}$ 0·9666. $n_D^{18\cdot8}$ 1·5084. Dipole moment $\times 10^{18} = 1{\cdot}0$.
$C_8H_{10}O,SbCl_3$: m.p. 42·2°.
$C_8H_{10}O,SbBr_3$: m.p. 48·8°.

Rodionov, *Bull. soc. chim.*, 1929, **45**, 109.
Zerbe, D.R.P., 563,969, (*Chem. Abstracts*, 1933, **27**, 4812).
Finzi, *Chem. Abstracts*, 1925, **19**, 2648.
King, Wright, *J. Chem. Soc.*, 1939, 1168.
Olson *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2451.
Hindley, Low, B.P. 588,806, (*Chem. Abstracts*, 1947, **41**, 6893).
Tyrer, B.P. 591,896, (*Chem. Abstracts*, 1948, **42**, 926).

Phenetole-2 : 5-disulphonic Acid

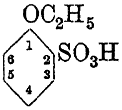


$C_8H_{10}O_7S_2$ MW, 282

Dichloride : $C_8H_8O_5Cl_2S_2$. MW, 319. Plates from Et_2O. M.p. 106–8°.
Diamide : $C_8H_{12}O_5N_2S_2$. MW, 280. Needles from H_2O. M.p. 233°.

Zander, *Ann.*, 1879, **198**, 25.

Phenetole-o-sulphonic Acid



$C_8H_{10}O_4S$ MW, 202

Cryst. mass.
Chloride : $C_8H_9O_3ClS$. MW, 220·5. Plates from pet. ether. M.p. 65–6°.
Amide : $C_8H_{11}O_3NS$. MW, 201. Needles from H_2O. M.p. 163°.
Anilide : m.p. 158°.
Phenylhydrazide : m.p. 132–3°.

Moody, *Chem. News*, 1893, **67**, 35.
Gattermann, *Ber.*, 1899, **32**, 1154.

Phenetole-m-sulphonic Acid.
Cryst. Sol. H_2O, EtOH.
Et ester : $C_{10}H_{14}O_4S$. MW, 230. Oil. Decomp. on dist. Volatile in steam.
Chloride : needles from Et_2O. M.p. 38°. Sol. C_6H_6, $CHCl_3$. Spar. sol. EtOH.
Amide : needles from H_2O. M.p. 131° (126°).

Lagai, *Ber.*, 1892, **25**, 1836.

Phenetole-p-sulphonic Acid.
Chloride : prisms or leaflets from Et_2O. M.p. 39° (36·5°). Sol. EtOH, Et_2O.
Amide : needles from H_2O, plates from EtOH. M.p. 150° (149°).

Lagai, *Ber.*, 1892, **25**, 1837.
Moody, *Chem. News*, 1892, **65**, 247.
Gattermann, *Ber.*, 1899, **32**, 1155.

p-Phenetylurea.
See Dulcin.

Phenmorpholone.
See under 2-Aminophenoxyacetic Acid.

Phenobarbital.
See Luminal.

Phenocoll.
See under *p*-Phenetidine.

Phenol (*Hydroxybenzene, carbolic acid*)

C_6H_5OH

C_6H_6O MW, 94

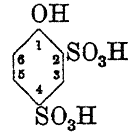
Needles. F.p. 41° (40·5°). M.p. 43°. B.p. 182°, 159·7°/400 mm., 90·2°/25 mm. Sol. EtOH, Et_2O. Mod. sol. H_2O. D_4^{25} 1·0710. n_D^{21} 1·5509. $k = 1{\cdot}3 \times 10^{-10}$ at 25°. $FeCl_3 \longrightarrow$ violet col. Turns pink in air. Corrosive poison. Heat of comb. C_p 732·5 Cal., C_v 734·2 (731–9) Cal. Volatile in steam.

$C_6H_6O,\frac{1}{2}H_2O$: m.p. 16°.
$C_6H_6O,2SbCl_3$: m.p. 37°.
$C_6H_6O,2SbBr_3$: m.p. 66·5°.
$2C_6H_6O,CH_3{\cdot}CO{\cdot}CH_3$: m.p. 15°.
$2C_6H_6O,CH_3{\cdot}CO{\cdot}NH_2$: m.p. 40·8°.
$C_6H_6O,CCl_3{\cdot}COOH$: m.p. 37·6°.
$2C_6H_6O,CO(NH_2)_2$: m.p. 61°.
$C_6H_6O,2CH_3{\cdot}NH_2$: m.p. 8·5–9·5°.
Picrate : m.p. 83·1°.
Phenylurethane : needles from C_6H_6. M.p. 126°. Dist. $\longrightarrow$ phenyl isocyanate. $NH_3 \longrightarrow$ phenylurea.
1-*Naphthylurethane* : m.p. 136–7°.
Sulphite : $C_{12}H_{10}O_3S$. MW, 234. B.p. 185°/15 mm., 143°/0·7 mm.
Hydrogen sulphate : *see* Phenyl hydrogen sulphate.
Hydrogen phosphite : $C_{12}H_{11}O_3P$. MW, 234. B.p. 218–19°/25 mm.
Phosphite : triphenyl phosphite. $C_{18}H_{15}O_3P$. MW, 310. B.p. 360°, 235°/18 mm.
Dihydrogen phosphate : $C_6H_7O_4P$. MW, 174. M.p. 99·5° (97–8°).
Hydrogen phosphate : diphenyl phosphate. $C_{12}H_{11}O_4P$. MW, 250. Needles or leaflets $+ 2H_2O$. M.p. 51°, anhyd. 70°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, hot H_2O.
Phosphate : *see* Triphenyl phosphate.
Orthosilicate : $C_{24}H_{20}O_4Si$. MW, 400. M.p. 47–8°. B.p. 417–20°.
Borate : triphenyl borate. $C_{18}H_{15}O_3B$. MW, 290. Needles from pet. ether. M.p. 50°.
Carbonate : *see* Diphenyl carbonate.
d-α-*Glucoside* : $C_{12}H_{16}O_6$. MW, 256. M.p. 174–5°. $[\alpha]_D^{20} - 71°$ in H_2O.
d-β-*Glucoside* : m.p. 139–41°. $[\alpha]_D^{20} - 39{\cdot}85°$ in H_2O.

Cappelli, *Gazz. chim. ital.*, 1918, **48**, ii, 107.
Vorozhtzov, Oshuev, *Chem. Abstracts*, 1934, **28**, 1027 (*Review*).
I.G., D.R.P., 613,726, (*Chem. Abstracts*, 1935, **29**, 8298).
du Pont, U.S.P., 2,007,327, *ibid.*, 5864.
Rütgerswerke A.-G., B.P. 427,145, *ibid.*, 6252.
Eikhman, Shemyakin, Vozhdaeva, *ibid.*, 2520.
Tishchenko, Churbakow, *ibid.*, 2520.
Noller, Dutton, *J. Am. Chem. Soc.*, 1933, **55**, 424.
Mizoshita, *Chem. Abstracts*, 1931, **25**, 1812.
Dyson, *Chemical Age*, 1926, **14**, 70 (*Review*).
Battegay, Denivelle, *Compt. rend.*, 1931, **192**, 492.
Bernton, *Ber.*, 1922, **55**, 3361.
Sultanov, *J. Gen. Chem. U.S.S.R.*, 1946, **16**, 1835, (*Chem. Abstracts*, 1947, **41**, 6224).

Phenoldiazonium chloride.
See Hydroxybenzenediazonium chloride.

Phenol-2 : 4-disulphonic Acid



$C_6H_6O_7S_2$ MW, 254

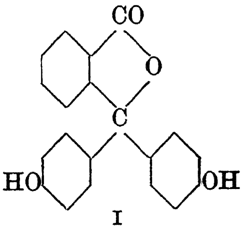
Needles. Decomp. above 100°. Sol. H_2O, EtOH. Insol. Et_2O.
Dichloride : $C_6H_4O_5Cl_2S_2$. MW, 291. Plates from Et_2O. M.p. 89° (86°).
Diamide : $C_6H_8O_5N_2S_2$. MW, 252. Leaflets. M.p. 239°.
Dianilide : m.p. 205°.

Gebauer-Fulnegg, Riesz, Austrian P., 119,960 (*Chem. Abstracts*, 1931, **25**, 1262).

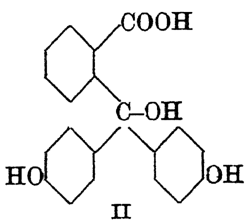
Phenol-2 : 5-disulphonic Acid.
Et ether : phenetole-2 : 5-disulphonic acid. *Dichloride* : $C_8H_8O_5Cl_2S_2$. MW, 319. Plates from Et_2O. M.p. 106–8°. *Diamide* : $C_8H_{12}O_5N_2S_2$. MW, 280. Needles from H_2O. M.p. 233°.

Senhofer, *Sitzb. Akad. Wiss. Wien*, 1878, **78**, ii, 678.

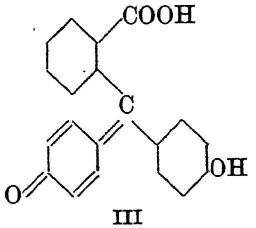
Phenolphthalein (3 : 3-*Di*-[p-*hydroxyphenyl*]-*phthalide*, α-4 : 4′-*trihydroxytriphenylmethane*-2-*carboxylic acid lactone*)



I



II



III

I. $C_{20}H_{14}O_4$ MW, 318
II. $C_{20}H_{16}O_5$ MW, 336
III. $C_{20}H_{14}O_4$ MW, 318

I.
Cryst. M.p. 254° (250–3°). Mod. sol. hot H_2O. Sol. hot Et_2O. NaOH ⟶ red col. Used as indicator. Red. ⟶ phenolphthalin.

Me ether: $C_{21}H_{16}O_4$. MW, 332. Needles from C_6H_6. M.p. 198–9° ⟶ glassy mass, m.p. 80°.

Di-Me ether: $C_{22}H_{18}O_4$. MW, 346. Leaflets from EtOH. M.p. 101–2° (97–9°). Sol. hot EtOH.

Di-Et ether: $C_{24}H_{22}O_4$. MW, 374. Leaflets from EtOH. M.p. 122° (118–20°). Sol. C_6H_6.

Diphenyl ether: $C_{32}H_{22}O_4$. MW, 470. Grey cryst. M.p. 105–6°.

Dibenzyl ether: $C_{34}H_{26}O_4$. MW, 498. Leaflets. M.p. 150°. Sol. C_6H_6.

Diacetyl: m.p. 143°.

Di-isovaleryl: m.p. 110°.

Dibenzoyl: m.p. 169°.

Dibenzenesulphonyl: m.p. 112–13°.

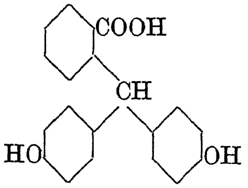
III.

Me ester: $C_{21}H_{16}O_4$. MW, 332. Orange-red needles. M.p. 127–30°. Sol. EtOH, $CHCl_3$.

Et ester: *Et ether*, $C_{24}H_{22}O_4$. MW, 374. Yellow cryst. M.p. 98–104°.

Underwood, Barker, *J. Am. Chem. Soc.*, 1930, **52**, 4084.
Molle, *Chem. Abstracts*, 1927, **21**, 2470 (*Bibl.*).
Day, *J. Am. Chem. Soc.*, 1930, **52**, 646.
Lund, *J. Chem. Soc.*, 1930, 1844.
Blicke, Swisher, *J. Am. Chem. Soc.*, 1934, **56**, 902.
Kavalco, U.S.P.s, 1,940,146, 1,940,494, (*Chem. Abstracts*, 1934, **28**, 1366).
Zelinskii, Maksorov, *Ind. Eng. Chem.*, 1931, **24**, 63.

Phenolphthalin (4 : 4′-*Dihydroxytriphenylmethane-2-carboxylic acid*)



$C_{20}H_{16}O_4$ MW, 320

Needles from H_2O. M.p. 225°. Spar. sol. H_2O. Ox. ⟶ phenolphthalein.

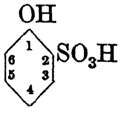
Et ester: $C_{22}H_{20}O_4$. MW, 348. Leaflets or needles from EtOH.Aq. M.p. 150–2° (156–8°).

Di-Me ether: $C_{22}H_{20}O_4$. MW, 348. Needles from EtOH. M.p. 149–50°.

Diacetyl: m.p. 146°.

Baeyer, *Ann.*, 1880, **202**, 80.

Phenol-*o*-sulphonic Acid



$C_6H_6O_4S$ MW, 174

Cryst. + $\frac{3}{4}H_2O$, partly melts at 50° decomp.: + H_2O, m.p. 145°. Sol. H_2O.

K salt, $2H_2O$: m.p. 235–40°.

Aniline salt: m.p. 165°.

Hexamethylenetetramine salt: m.p. 140°. B.p. 180°.

Di-hexamethylenetetramine salt: m.p. 160°.

Chloride: *acetyl deriv.*, cryst. from ligroin. M.p. 72·2–72·9°. Sol. EtOH, C_6H_6.

Anilide: m.p. 126·5–127·5°. *Acetyl*, m.p. 106–7°.

p-*Toluidide*: m.p. 124–5°. *Acetyl*, m.p. 116–17°.

Tomcsik, *Chem. Abstracts*, 1930, **24**, 4517.
Anschütz, *Ann.*, 1918, **415**, 68.

Phenol-*m*-sulphonic Acid.

Needles + $2H_2O$. $FeCl_3$ ⟶ violet col.

K salt, H_2O: m.p. 200–10°.

Chloride: $C_6H_5O_3ClS$. MW, 192·5. Brown oil. Sol. H_2O, EtOH, Et_2O.

Obermiller, *Ann.*, 1911, **381**, 115.
Szathmary, *Ber.*, 1910, **43**, 2485.

Phenol-*p*-sulphonic Acid.

Ammonium salt: m.p. 270–1° decomp.

Sulphanilamide salt: m.p. 241–2°.

Aniline salt: m.p. 170°.

Hexamethylenetetramine salt: m.p. 180–2°.

Di-hexamethylenetetramine salt: m.p. about 190°.

S-*Benzylisothiouronium salt*: (+ H_2O), m.p. 168·7°.

p-*Nitrobenzylpyridinium salt*: m.p. 162°.

Et ester: *benzoyl deriv.*, m.p. 62°.

Fluoride: $C_6H_5O_3FS$. MW, 176. M.p. 77°.

Chloride: $C_6H_5O_3ClS$. MW, 192·5. *Acetyl*, m.p. 78°. B.p. 148°/12. *Carbethoxyl*, m.p. 75°. *Benzoyl*, m.p. 115–16°.

Amide: $C_6H_7O_3NS$. MW, 173. Cryst. from EtOH. M.p. 176–7°.

Anilide: m.p. 141°. *Acetyl*, m.p. 126–7°. *Aniline salt*: m.p. 112–13°.

p-*Toluidide*: m.p. 151–2°. *Acetyl*, m.p. 98–9°.

Piperidide: m.p. 132–3°. *Acetyl*, m.p. 115–16°.

Major, B.P. 328,220, (*Chem. Abstracts*, 1930, **24**, 5509).
Steinkopf, D.R.P., 497,242, (*Chem. Abstracts*, 1930, **24**, 3517).
Fichter, Tamm, *Ber.*, 1910, **43**, 3036.
Anschütz, Molineus, *Ann.*, 1918, **415**, 56.

Phenol-tricarboxylic Acid.

See 5-Hydroxytrimellitic Acid *and* Hydroxytrimesic Acid.

Phenomydrol.

See Phenacylamine.

Phenoprene.

See 2-Phenyl-1 : 3-butadiene.

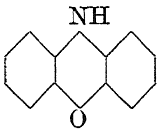
Phenothiazine.

See Thiodiphenylamine.

Phenothioxine.
See Phenoxthine.

Phenoxacetin.
See under Glycerol.

Phenoxazine (*Phenazoxine*)



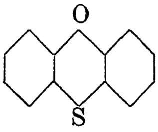
$C_{12}H_9ON$ MW, 183

Leaflets from EtOH. M.p. 156° (153–4°). $FeCl_3$ ⟶ bluish-green col. H_2SO_4 ⟶ violet-red col.

Acetyl deriv.: prisms. M.p. 142°.

Cullinane, Davey, Padfield, *J. Chem. Soc.*, 1934, 718.
Kehrmann, Neil, *Ber.*, 1903, **47**, 3107.
Fresenius, *Z. anal. Chem.*, 1934, **96**, 433 (*Review*).
Waterman, Vivian, U.S.P. 2,292,808, (*Chem. Abstracts*, 1943, **37**, 892).

Phenoxthine (*Phenothioxine, dibenzthioxine*)



$C_{12}H_8OS$ MW, 200

Cryst. from MeOH. M.p. 59° (57·5–58°). B.p. 185–7°/23 mm. H_2SO_4 ⟶ intense violet col. Reagent for traces of Pd ⟶ yellow col. in absence of Au.

Suter, McKenzie, Maxwell, *J. Am. Chem. Soc.*, 1936, **58**, 717.
A.G.F.A., D.R.P., 234,743, (*Chem. Abstracts*, 1911, **5**, 2912).
Suter, Maxwell, *Organic Syntheses*, 1938, XVIII, 64.
Ferrario, *Bull. soc. chim.*, 1911, **9**, 536.

Phenoxyacetaldehyde (*ω-Aldehydoanisole, glycollic aldehyde phenyl ether*)

$C_6H_5O{\cdot}CH_2{\cdot}CHO$

$C_8H_8O_2$ MW, 136

Cryst. + $1H_2O$. M.p. 38°. B.p. 215° decomp., 118–19°/30 mm., 101–3°/15 mm. Unstable. D_4^{21} 1·1310. n_D^{21} 1·5380.

Oxime: m.p. 95°.

Semicarbazone: m.p. 145°.

Phenylhydrazone: m.p. 86°.

Di-Et acetal: b.p. 257°, 136–8°/16 mm. D_4^{20} 1·0183. n_D^{20} 1·4878.

Rotbart, *Ann. chim.*, 1934, **1**, 479.
Hatch, Nesbitt, *J. Am. Chem. Soc.*, 1945, **67**, 39.
Yamashita, Matsumura, *Chem. Abstracts*, 1947, **41**, 3753.

Phenoxyacetic Acid (*Glycollic acid phenyl ether*)

$C_6H_5O{\cdot}CH_2{\cdot}COOH$

$C_8H_8O_3$ MW, 152

Needles from H_2O. M.p. 98–9°. B.p. 285° decomp. Sol. EtOH, Et_2O, AcOH, C_6H_6, CS_2. $k = 7{\cdot}56 \times 10^{-4}$ at 25°.

o-*Phenylenediamine salt*: m.p. 137°.

m-*Phenylenediamine salt*: m.p. 125–6°.

Di-p-*phenylenediamine salt*: m.p. 148–9° decomp.

Me ester: $C_9H_{10}O_3$. MW, 166. B.p. 245°. $D^{17.5}$ 1·150.

Et ester: $C_{10}H_{12}O_3$. MW, 180. B.p. 251°, 145–50°/30 mm. $D^{17.5}$ 1·104.

Phenyl ester: $C_{14}H_{12}O_3$. MW, 228. M.p. 56°. B.p. 320–5°, 236°/73 mm., 185°/30 mm. (196°/13 mm.).

Chloride: $C_8H_7O_2Cl$. MW, 170·5. B.p. 225–6°, 165°/60 mm., 111°/13 mm., 109°/9 mm.

Amide: $C_8H_9O_2N$. MW, 151. Needles from H_2O. M.p. 101·5°.

Anhydride: $C_{16}H_{14}O_5$. MW, 286. Leaflets from Et_2O. M.p. 67–9°.

Nitrile: C_8H_7ON. MW, 133. B.p. 239–40°. $D^{17.5}$ 1·09.

Anilide: m.p. 101·5° (99°).

N-*Benzylamide*: m.p. 84·5–86°.

Monsanto, U.S.P., 1,974,810, (*Chem. Abstracts*, 1934, **28**, 7262).
van Alphen, *Rec. trav. chim.*, 1927, **46**, 144.
Blaise, Picard, *Ann. chim.*, 1912, **26**, 274.

Phenoxyacetone (*Phenyl acetonyl ether, acetol phenyl ether, ω-acetoanisole*)

$C_6H_5O{\cdot}CH_2{\cdot}CO{\cdot}CH_3$

$C_9H_{10}O_2$ MW, 150

Oil. B.p. 229–30°.

Semicarbazone: m.p. 173°.

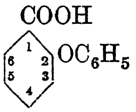
Guanylhydrazone: m.p. 154°.

St. Wehln, *Ber.*, 1902, **35**, 3553 (*Footnote*).
Stoermer, *Ber.*, 1895, **28**, 1253.

Phenoxyanthraquinone.
See under Hydroxyanthraquinone.

4-Phenoxyazobenzene.
See under 4-Hydroxyazobenzene.

***o*-Phenoxybenzoic Acid** (*Salicylic acid phenyl ether, diphenyl ether 2-carboxylic acid*)



$C_{13}H_{10}O_3$ MW, 214

Leaflets from EtOH.Aq. M.p. 113°. B.p. 355°. Sol. EtOH, Et_2O. Spar. sol. hot H_2O. Dist. ⟶ trace of xanthone.

Me ester: $C_{14}H_{12}O_3$. MW, 228. B.p. 312°.

Et ester: $C_{15}H_{14}O_3$. MW, 242. B.p. above 360°.

Phenyl ester: $C_{19}H_{14}O_3$. MW, 290. Needles from EtOH. M.p. 109°.

Amide: $C_{13}H_{11}O_2N$. MW, 213. Prisms from EtOH. M.p. 131°. Sol. ord. org. solvents.

A.G.F.A., D.R.P., 158,998, (*Chem. Zentr.*, 1905, I, 843).
Ullmann, Zlokasoff, *Ber.*, 1905, **38**, 2117.
Knapp, U.S.P. 2,251,743, (*Chem. Abstracts*, 1941, **35**, 7420).
Langham, Brewster, Gilman, *J. Am. Chem. Soc.*, 1941, **63**, 545.

***m*-Phenoxybenzoic Acid** (m-*Hydroxybenzoic acid phenyl ether, diphenyl ether* 3-*carboxylic acid*).

Needles from EtOH.Aq. M.p. 145°. Sol. EtOH, Et_2O. Insol. H_2O.

Griess, *Ber.*, 1888, **21**, 980.
Langham, Brewster, Gilman, *J. Am. Chem. Soc.*, 1941, **63**, 545.
Willis, *Chem. Abstracts*, 1944, **38**, 740.

***p*-Phenoxybenzoic Acid** (p-*Hydroxybenzoic acid phenyl ether, diphenyl ether* 4-*carboxylic acid*).

Prisms from $CHCl_3$. M.p. 159·5°. Sol. EtOH, Et_2O.

Phenyl ester: cryst. from EtOH.Aq. M.p. 73–8°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

Klein, *J. prakt. Chem.*, 1883, **28**, 199.
Griess, *Ber.*, 1888, **21**, 980.
Schönberg, Kraemer, *Ber.*, 1922, **55**, 1190.
Gilman, Langham, Moore, *J. Am. Chem. Soc.*, 1940, **62**, 2327.
Langham, Brewster, Gilman, *J. Am. Chem. Soc.*, 1941, **63**, 545.

2-*p*-Phenoxybenzoylbenzoic Acid.

See under 4′-Hydroxybenzophenone-2-carboxylic Acid.

1-Phenoxybutyric Acid

$CH_3{\cdot}CH_2{\cdot}CH(OC_6H_5){\cdot}COOH$

$C_{10}H_{12}O_3$ MW, 180

Needles from H_2O, plates from ligroin. M.p. 82–3° (99°). B.p. 258°. Sol. ord. org. solvents. $k = 6{\cdot}82 \times 10^{-4}$.

Ag salt: m.p. 202° decomp.

Et ester: $C_{12}H_{16}O_3$. MW, 208. B.p. 250–1°/748 mm., 175–80°/41 mm. Sol. EtOH, Et_2O.

Phenyl ester: $C_{16}H_{16}O_3$. MW, 256. Prisms from MeOH. M.p. 48–9°. B.p. 202–3°/25 mm. D_{15}^{15} 1·136.

Chloride: $C_{10}H_{11}O_2Cl$. MW, 198·5. B.p. 128–31°/38 mm.

Amide: $C_{10}H_{13}O_2N$. MW, 179. M.p. 111° (123°). Sol. Et_2O, Me_2CO, $CHCl_3$, C_6H_6, hot EtOH.

Nitrile: $C_{10}H_{11}ON$. MW, 161. B.p. 228–30°/748 mm. decomp.

Anilide: m.p. 93–4°.

Bischoff, *Ber.*, 1900, **33**, 931; 1901, **34**, 1837.

3-Phenoxybutyric Acid

$C_6H_5O{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_{10}H_{12}O_3$ MW, 180

Leaflets from ligroin. M.p. 64–5° (60°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. CS_2, ligroin. Insol. H_2O.

Et ester: b.p. 170–3°/25 mm. D_{25}^{33} 1·048. n_D^{33} 1·491.

Amide: leaflets from EtOH.Aq. M.p. 80°.

Nitrile: needles. M.p. 45–6°. B.p. 287–9°/765 mm.

Bentley, Haworth, Perkin, *J. Chem. Soc.*, 1896, **69**, 168.
Putochin, *Ber.*, 1922, **55**, 2747.
Nair, Peacock, *J. Indian Chem. Soc.*, 1935, **12**, 318.
Marvel, Tanenbaum, *J. Am. Chem. Soc.*, 1922, **44**, 2647.

Phenoxycyclohexane.

See Phenyl cyclohexyl Ether.

α-Phenoxydi-1-naphthylmethane.

See under 1 : 1′-Dinaphthylcarbinol.

Phenoxyethyl Alcohol.

See 2-Hydroxyethyl phenyl Ether.

1-Phenoxyisobutyric Acid

$(CH_3)_2C(OC_6H_5){\cdot}COOH$ [printed as CH_3, CH_3 > C < OC_6H_5, COOH]

$C_{10}H_{12}O_3$ MW, 180

Needles from H_2O. M.p. 97–8° (98–9°). Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, AcOH. Mod. sol. hot H_2O. $k = 4{\cdot}34 \times 10^{-4}$. Volatile in steam.

Et ester: $C_{12}H_{16}O_3$. MW, 208. B.p. 243°, 127°/12 mm.

Phenyl ester: $C_{16}H_{16}O_3$. MW, 256. Needles. M.p. 24–6°. B.p. 194–6°/16 mm.

Chloride: $C_{10}H_{11}O_2Cl$. MW, 198·5. B.p. 112–13°/12·5 mm. (impure).

Amide: $C_{10}H_{13}O_2N$. MW, 179. Needles from EtOH. M.p. 116° (114°).

Bischoff, *Ber.*, 1900, **33**, 933; 1901, **34**, 1837.
Link, D.R.P., 80,986.
Gabriel, *Ber.*, 1913, **46**, 1347 (*Footnote*).
Bargellini, *Gazz. chim. ital.*, 1906, **36**, ii, 334.
Nazarov, Kuznetsova, *Chem. Abstracts*, 1942, **36**, 1298.

1 - Phenoxynaphthalene - 8 - carboxylic Acid.

See under 8-Hydroxy-1-naphthoic Acid.

α-Phenoxyphenylacetic Acid.

See under Mandelic Acid.

1-Phenoxypropionic Acid (*Lactic acid phenyl ether*)

$CH_3{\cdot}CH(OC_6H_5){\cdot}COOH$

$C_9H_{10}O_3$ MW, 166

Needles from H_2O. M.p. 115–16° (112–13°). B.p. 265–6°/758 mm. Sol. EtOH, Et_2O, hot H_2O. $k = 7{\cdot}75 \times 10^{-4}$. Volatile in steam.

Et ester: $C_{11}H_{14}O_3$. MW, 194. B.p. 243–4°, 160–4°/50 mm., 120–5°/6 mm. $D^{17\cdot5}$ 1·360.

Di-methylaminoethyl ester: $C_{13}H_{19}O_3N$. MW, 237. M.p. 75°. B.p. 116°/0·2 mm. *Picrate*: m.p. 113°. *Picrolonate*: m.p. 148°.

Phenyl ester: $C_{15}H_{14}O_3$. MW, 242. M.p. 52°. B.p. 190°/18 mm. D_{15}^{15} 1·147.

Tropine ester: $C_{17}H_{23}O_3N$. MW, 289. Yellow oil. B.p. 149–50°/0·6 mm. *Picrate*: m.p. 175°. *Picrolonate*: m.p. 200–2°.

Chloride: $C_9H_9O_2Cl$. MW, 184·5. B.p. 146–7°/55 mm., 120°/30 mm., 115°/20 mm., (115–17°/10 mm.).

Amide: $C_9H_{11}O_2N$. MW, 165. Needles from H_2O. M.p. 132–3° (130°). Sol. EtOH, Et_2O, AcOH, CS_2, hot ligroin.

Diphenylamide: m.p. 93°.

Anilide: m.p. 117° (118–19°). B.p. 211–12°/14 mm.

Me-anilide: m.p. 57·5°. B.p. 206°/18 mm.

Et-anilide: m.p. 47·5°. B.p. 224–5°/17 mm.

o-*Nitroanilide*: m.p. 88°. B.p. 248°/28 mm.

m-*Nitroanilide*: m.p. 118°.

p-*Nitroanilide*: m.p. 141–2°.

o-*Toluidide*: m.p. 88–90°. B.p. 220–30°/24 mm.

m-*Toluidide*: m.p. 86·5°. B.p. 220°/15 mm.

p-*Toluidide*: m.p. 115°.

Phenylbenzylamide: m.p. 111–12°.

1-*Naphthylamide*: m.p. 131°. B.p. 260°/20 mm.

2-*Naphthylamide*: m.p. 117°.

Plazek, Rodewald, Krzyzaniak, *Chem. Zentr.*, 1936, I, 1212.

Fourneau, Sandulesco, *Bull. soc. chim.*, 1922, **31**, 988.

2-Phenoxypropionic Acid (*Hydracrylic acid phenyl ether*)

$C_6H_5O{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_9H_{10}O_3$ MW, 166

M.p. 97·5–98°. B.p. 235–45°/771 mm. $k = 5{\cdot}4 \times 10^{-5}$.

Me ester: b.p. 106–19°/5–10 mm.

Powell, *J. Am. Chem. Soc.*, 1923, **45**, 2708.

Bischoff, *Ber.*, 1900, **33**, 928.

Ufer, D.R.P., 670,357.

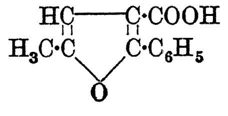
Phenoxypropylene oxide.

See under Glycide.

Phenthiazine.

See Thiodiphenylamine.

Phenuvic Acid (5-*Methyl*-2-*phenylfuran*-3-*carboxylic acid*, 5-*methyl*-2-*phenyl*-β-*furoic acid*)



$C_{12}H_{10}O_3$ MW, 202

Needles from EtOH.Aq. M.p. 147–8° (144–5°). Sol. EtOH, C_6H_6, ligroin. Spar. sol. hot H_2O.

Et ester: $C_{14}H_{14}O_3$. MW, 230. Oil. B.p. 193–4°/20 mm.

Borsche, Fels, *Ber.*, 1906, **39**, 1923, 1927.

Phenylacetaldehyde (α-*Toluic aldehyde*)

$C_6H_5{\cdot}CH_2{\cdot}CHO$

C_8H_8O MW, 120

Cryst. from H_2O. M.p. 33–4°. B.p. 195°, 88°/18 mm., 78°/10 mm. $D_4^{19\cdot6}$ 1·0272. $n_D^{19\cdot6}$ 1·5255. Cold 10% KOH ⟶ dimer.

Dimer: amorph. M.p. 50°. Sol. most org. solvents. Depolymerises at 100°.

Cyanhydrin: *see under* α-Hydroxyhydrocinnamic Acid.

Di-Me acetal: oil. B.p. 219–21°/754 mm. D^{18} 1·0042.

Di-Et acetal: b.p. 245–6°.

Oxime: m.p. 98·5°.

Phenylhydrazone: prisms from ligroin. M.p. 62–3°.

Diphenylhydrazone: cryst. from EtOH. M.p. 101–2°.

2 : 4-*Dinitrophenylhydrazone*: golden leaflets from EtOH. M.p. 110° (240°).

Erlenmeyer, Lipp, *Ann.*, 1883, **219**, 182.

Rassow, Burmeister, *J. prakt. Chem.*, 1911, **84**, 487.

Wood, Cowley, *J. Soc. Chem. Ind.*, 1923, **42**, 429T.

Kodama, *Chem. Abstracts*, 1922, **16**, 106.

Yamashita, Matsumura, *J. Chem. Soc. Japan*, 1943, **64**, 506, (*Chem. Abstracts*, 1947, **41**, 3753).

Isagulyants, Russ. P. 46,918, (*Chem. Abstracts*, 1939, **33**, 3399).

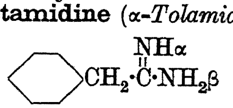
Shumeiko, *J. Applied Chem. U.S.S.R.*, 1941, **14**, 93, (*Chem. Abstracts*, 1942, **36**, 436).

Guseva, *Chem. Abstracts*, 1942, **36**, 3794.

Phenylacetamidoacetaldehyde.

See Penilloaldehyde-G.

Phenylacetamidine (α-*Tolamidine*)



$C_8H_{10}N_2$ MW, 134

Benzenesulphonate: m.p. 186–186·5°.

β-N-*Me*: oil. *Reineckate*: plates from Me_2CO.Aq. M.p. about 137°.

β-N-*Benzyl*: m.p. 93°. *Benzenesulphonate*: m.p. 131°.

β-N-p-*Tolyl*: m.p. 119°.

β-N-o-*Chlorophenyl*: m.p. 111·5–112°. *Picrate*: m.p. 153–153·5°.

Oxley, Short, *J. Chem. Soc.*, 1947, 147.

***N*-Phenylacetanilide.**

See under Diphenylamine.

Phenyl acetate

$CH_3 \cdot CO \cdot OC_6H_5$

$C_8H_8O_2$ MW, 136

B.p. 195·7°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O. D_4^0 1·0927, D_{15}^{15} 1·0809.

Hoeflake, *Rec. trav. chim.*, 1916, **36**, 30.

N-Phenylacethydrazide.

See under Phenylhydrazine.

Phenylacetic Acid (*α-Toluic acid, toluylic acid*)

$C_6H_5 \cdot CH_2 \cdot COOH$

$C_8H_8O_2$ MW, 136

Plates from pet. ether. M.p. 76–76·5°. B.p. 265·5°, 144·2–144·8°/12 mm. D_4^{77} 1·091. Sol. EtOH, Et_2O. Spar. sol. cold H_2O. $k = 5{\cdot}56 \times 10^{-5}$ at 25°. Heat of comb. C_v 930·7 Cal.

Me ester: $C_9H_{10}O_2$. MW, 150. B.p. 215°, 131–2°/50 mm. D_0^0 1·0808, D_{16}^{16} 1·0633. n_D^{16} 1·5091.

Et ester: $C_{10}H_{12}O_2$. MW, 164. B.p. 227·1–227·6°, 132–8°/32 mm., 120–5°/17–18 mm. D_4^{20} 1·0333. $n_D^{18.5}$ 1·49921. Used in perfumery.

Propyl ester: $C_{11}H_{14}O_2$. MW, 178. B.p. 238°. D^{18} 1·0142.

Isobutyl ester: $C_{12}H_{16}O_2$. MW, 192. B.p. 247°, 123–5°/14 mm.

tert.-*Butyl ester*: f.p. − 21°. B.p. 110°/15 mm. D_4^{25} 0·9758. n_D 1·4825.

d-*Amyl ester*: b.p. 265–6°/722·7 mm. D_4^{20} 0·982. n_D^{21} 1·4872. $[\alpha]_D^{22}$ + 4·92°.

l-*Menthyl ester*: liq. B.p. 216°/39 mm., 197°/15 mm., 94–5°/0·25 mm. D_4^{20} 0·9874. $[\alpha]_D^{16.5}$ − 68·15°.

Phenyl ester: $C_{14}H_{12}O_2$. MW, 212. Needles from EtOH.Aq. M.p. 42°. B.p. 158°/7 mm. Sol. EtOH, AcOH, pet. ether. Spar. sol. cold Et_2O. Insol. H_2O.

Benzyl ester: $C_{15}H_{14}O_2$. MW, 226. Oil. B.p. 317–19°, 270°/160 mm., 175–6°/12 mm. D^{17} 1·0938. Sol. EtOH, Et_2O. Spar. sol. cold H_2O.

p-*Nitrobenzyl ester*: m.p. 65°.

p-*Tolyl ester*: m.p. 75–6°.

Fluoride: phenacetyl fluoride. C_8H_7OF. MW, 138. Oil. B.p. 88–9°/17 mm.

Chloride: phenacetyl chloride. C_8H_7OCl. MW, 154·5. Liq. B.p. 170°/250 mm., 104–5°/24 mm., 100°/12 mm. D_4^4 1·1856, D_4^{20} 1·16817.

Bromide: phenacetyl bromide. C_8H_7OBr. MW, 199. B.p. 150–5°/50 mm.

Amide: phenacetamide. C_8H_9ON. MW, 135. Plates or leaflets. M.p. 157°. Sol. EtOH. Spar. sol. H_2O, Et_2O, C_6H_6. N-*Acetyl*: needles from EtOH.Aq. M.p. 129°. N-*Benzoyl*: prisms from C_6H_6, needles from EtOH. M.p. 129–30°.

Methylamide: cryst. from C_6H_6. M.p. 58°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O, C_6H_6.

Dimethylamide: cryst. M.p. 43·5°. B.p. 155°/10 mm. Sol. H_2O, Et_2O, $CHCl_3$, C_6H_6.

Ethylamide: plates from H_2O. M.p. 73–4°.

Diethylamide: leaflets. M.p. 86°. B.p. 297°.

Dipropylamide: oil. B.p. 183–4°/16 mm.

Benzylamide: m.p. 122°.

Nitrile: *see* Benzyl cyanide.

Anhydride: $C_{16}H_{14}O_3$. MW, 254. Needles. M.p. 72°.

Anilide: prisms from EtOH. M.p. 117–18°. Sol. EtOH, Et_2O.

o-*Toluidide*: needles from C_6H_6. M.p. 159°. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. Et_2O, CS_2, ligroin.

p-*Toluidide*: leaflets from EtOH. M.p. 135–6°. Mod. sol. EtOH, Et_2O.

Hydrazide: needles from H_2O. M.p. 116°. Sol. warm EtOH, H_2O. Mod. sol. hot Et_2O. Reduces warm Fehling's. *B,HCl*: needles. M.p. 215° decomp. Sol. H_2O.

Phenylhydrazide: m.p. 173–4°.

Adams, Thal, *Organic Syntheses*, Collective Vol. I, 265, 427.
Heilbron, Hey, Lythgoe, *J. Chem. Soc.*, 1936, 297.
Sobin, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 2458.
Yabroff, Porter, *J. Am. Chem. Soc.*, 1932, **54**, 2453.
Kindler, Giese, Hesse, *Arch. Pharm.*, 1927, **265**, 389.
Wieland, Fischer, *Ann.*, 1925, **446**, 65.
Autenrieth, Thomae, *Ber.*, 1924, **57**, 431.
Adams, Ulich, *J. Am. Chem. Soc.*, 1920, **42**, 599.
Traube, Krahmer, *Ber.*, 1919, **52**, 1296.
Taverne, *Rec. trav. chim.*, 1897, **16**, 34.

1-Phenylacetoacetic Acid

$CH_3 \cdot CO \cdot CH(C_6H_5) \cdot COOH$

$C_{10}H_{10}O_3$ MW, 178

Et ester: $C_{12}H_{14}O_3$. MW, 206. Oil. B.p. 145–7°/11 mm. Mod. sol. alkalis. Alc. $FeCl_3$ ⟶ intense violet col.

l-*Menthyl ester*: needles from MeOH. M.p. 69°. B.p. 204–10°/10 mm., 131–3°/0·1 mm. Shows mutarotation.

Nitrile: $C_{10}H_9ON$. MW, 159. Cryst. from EtOH.Aq. or AcOEt–pet. ether. M.p. 90–1°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. hot H_2O, pet. ether. Volatile in steam. Alc. $FeCl_3$ ⟶ green col. *Phenylhydrazone*: needles from MeOH. M.p. 114°. *Anil*: leaflets from C_6H_6. M.p. 102–3°.

Anilide: needles from EtOH.Aq. M.p. 97°. Sol. EtOH. Spar. sol. H_2O, Et_2O.

Rupe, *Ann.*, 1913, **398**, 372.
Bodroux, *Bull. soc. chim.*, 1910, **7**, 851.
Beckh, *Ber.*, 1898, **31**, 3161.
Heller, Herrmann, Spielmeyer, *J. prakt. Chem.*, 1928, **120**, 196.
Kimball, Jefferson, Pike, *Organic Syntheses*, 1938, XVIII, 36.

3-Phenylacetoacetic Acid

$C_6H_5{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}COOH$

$C_{10}H_{10}O_3$ MW, 178

Me ester: b.p. 125°/3 mm.

Et ester: $C_{12}H_{14}O_3$. MW, 206. B.p. 153–5°/9 mm., 118–23°/0·6 mm. n_D^{25} 1·5066. MeOH + $FeCl_3$ ⟶ deep red col. Tetranitromethane in MeOH ⟶ faint yellowish-green col., without solvent ⟶ reddish-orange col. Does not reduce Fehling's. $NaNO_2$ ⟶ deep blue col. *Cu chelate*: m.p. 128–9°.

Cu salt: cryst. from C_6H_6. M.p. 176–8°. Sol. $CHCl_3$. Spar. sol. Et_2O.

Semicarbazone: needles from MeOH.Aq. M.p. 113–16°. Sol. EtOH. Insol. H_2O, Et_2O.

2 : 4-*Dinitrophenylhydrazone*: m.p. 155°.

Anil: needles from 75% MeOH. M.p. 96–98·5°. Sol. Et_2O, AcOEt. Spar. sol. cold MeOH, pet. ether, ligroin. Insol. H_2O.

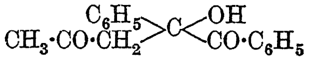
Sonn, Litten, *Ber.*, 1933, **66**, 1512.

Phenylacetol.
See 3-Phenylhydroxyacetone.

***p*-Phenylacetophenone.**
See 4-Acetodiphenyl.

Phenylacetone.
See Methyl benzyl Ketone.

Phenylacetonylbenzoylcarbinol (*Acetone-benzil*)



$C_{17}H_{16}O_3$ MW, 268

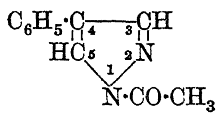
Prisms from Et_2O. M.p. 78°. Sol. Et_2O, hot EtOH. At 200° ⟶ benzil + acetone. Ox. ⟶ acetic + benzoic acids.

Monoxime: cryst. from EtOH. M.p. 146°. Sol. hot C_6H_6. Mod. sol. Et_2O.

Japp, Miller, *J. Chem. Soc.*, 1885, **47**, 24.

Phenyl acetonyl Ether.
See Phenoxyacetone.

4-Phenyl-1-acetopyrazole (*Acetylphenyl-pyrazole*)



$C_{11}H_{10}ON_2$ MW, 186

Pale yellow needles from MeOH or Et_2O. M.p. 81·5–82·5°. B.p. 159–61°/12 mm. $D_4^{99·9}$ 1·0945. n_D 1·5589.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 177.

1-Phenyl-3-acetopyrazole (3-*Acetyl*-1-*phenylpyrazole*).

Phenylhydrazone: yellow plates. M.p. 182°.

Diels, Petersen, *Ber.*, 1922, **55**, 3454.

1-Phenyl-4-acetopyrazole (4-*Acetyl*-1 *phenylpyrazole*).

Needles from EtOH.Aq. M.p. 121·5–122·5°. Sol. H_2O.

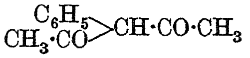
Oxime: needles from 50% EtOH. M.p. 129–31°. Spar. sol. H_2O.

Phenylhydrazone: needles from EtOH. M.p. 142–4° decomp.

Balbiano, *Gazz. chim. ital.*, 1889, **19**, 197.

***N*-Phenylaceturic Acid.**
See under Phenylglycine.

Phenylacetylacetone (3-*Phenylpentandione*-2 : 4)



$C_{11}H_{12}O_2$ MW, 176

Oil with odour of pine shavings. M.p. 58–60°. B.p. 127–8°/15 mm. $FeCl_3$ ⟶ purple col.

Cu salt: dark green cryst. Decomp. at 222–4°. Sol. usual org. solvents.

Morgan, Drew, Porter, *Ber.*, 1925, **58**, 333.
Adams, Hauser, *J. Am. Chem. Soc.*, 1945, **67**, 284.

Phenylacetylcarbinol (*Methyl α-hydroxy-benzyl ketone*, 1-*hydroxy*-1-*phenylacetone*)

$CH_3{\cdot}CO{\cdot}CH(OH){\cdot}C_6H_5$

$C_9H_{10}O_2$ MW, 150

Yellowish oil. B.p. 205–7°, 135–7°/24 mm., 140–5°/11 mm. Misc. with most org. solvents. Reduces $NH_3.AgNO_3$ and Fehling's.

Acetyl: pale yellow oil. B.p. 165–70°/40 mm.

Oxime: needles from H_2O. M.p. 112–15°.

Semicarbazone: powder from toluene. M.p. 194°. Sol. boiling AcOH. Spar. sol. other solvents.

2 : 4-*Dinitrophenylhydrazone*: orange needles from EtOH. M.p. 170°.

Note: phenylacetylcarbinol is tautomeric (keto-carbinol tautomerism) with methylbenzoylcarbinol (β-hydroxypropiophenone).

Hey, *J. Chem. Soc.*, 1930, 1232.
Meerwein, Hinz, *Ann.*, 1930, **484**, 1.
Favorskiĭ, Tennikova, *Compt. rend.*, 1934, **198**, 1998.
Freudenberg, *Biochem. Z.*, 1932, **245**, 238.
Roger, *Biochem. Z.*, 1931, **230**, 320.
Kotchergine, *Bull. soc. chim.*, 1928, **43**, 573.

Phenylacetyl-carbinol.
See 3-Phenylhydroxyacetone.

Phenylacetylene (*Acetylenylbenzene*, *ethynyl-benzene*)

$C_6H_5{\cdot}C⋮CH$

C_8H_6 MW, 102

Oil. F.p. −45°. B.p. 142–4°, 49–50°/14 mm. $D_4^{12·5}$ 0·9371. $n_D^{17·4}$ 1·5501. Heat of comb. C_v 1024·5 Cal.

Dibromide: *see* α : β-Dibromostyrene.

Dichloride : *see* α : β-Dichlorostyrene.

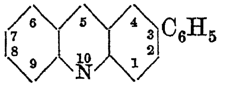
Vaughn, *J. Am. Chem. Soc.*, 1934, **56**, 2064.
Hessler, *Organic Syntheses*, Collective Vol. I, 428.
Bodzoux, *Compt. rend.*, 1939, **208**, 1022.
Sandhaas, U.S.P. 2,317,808, (*Chem. Abstracts*, 1943, **37**, 5987).

Phenylacetylene dichloride.
See αβ-Dichlorostyrene.

Phenylacetylglycine.
See Phenaceturic Acid.

2-Phenylacetyl-propionic Acid.
See 4-Phenyl-levulinic Acid.

3-Phenylacridine.



$C_{19}H_{13}N$ MW, 255

Plates. M.p. 127–8°.
Picrate : m.p. 244–5° decomp.

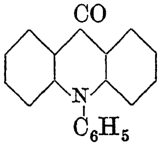
Petrow, *J. Chem. Soc.*, 1942, 693.

5-Phenylacridine (ms-*Phenylacridine*).
Pale yellow leaflets from EtOH. M.p. 184° (181°). B.p. 403–4°. Sol. C_6H_6. Mod. sol. Et_2O. Spar. sol. EtOH. Insol. H_2O. Sol. conc. H_2SO_4. Sols. in acids show green fluor. Sublimes in leaflets. Triboluminescent.
Picrate : cryst. from EtOH. M.p. 227·7°.
Dipicrate : cryst. from EtOH. M.p. 185–6°.
Methohydroxide : plates from C_6H_6. M.p. 140°. Sol. Et_2O, $CHCl_3$, C_6H_6, pet. ether. Insol. H_2O. Sol. acids.
Methochloride : cryst. + $2H_2O$. Decomp. at 225–6°. Very sol. H_2O, EtOH, $CHCl_3$, Py, $PhNO_2$. Insol. Et_2O, CCl_4.
Methobromide : greenish-yellow cryst. + H_2O. Decomp. at 230°. Sol. H_2O. Mod. sol. EtOH, $CHCl_3$, Py, $PhNO_2$, aniline.
Methiodide : black prisms from EtOH. Sol. EtOH, $CHCl_3$, Py, $PhNO_2$. Less sol. H_2O, Me_2CO, C_6H_6. Insol. Et_2O, CCl_4.
Methonitrate : yellow needles. Decomp. at 288°. Sol. EtOH, $CHCl_3$.
Ethohydroxide : cryst. from toluene. M.p. 136–7°. Sol. Et_2O, Me_2CO, $CHCl_3$.
Ethiodide : dark red needles from H_2O. M.p. 223° decomp.
Ethopicrate : yellow needles from EtOH. M.p. 181°.
Phenohydroxide : prisms from ligroin. M.p. 178°. Sol. Et_2O, EtOH, C_6H_6, hot ligroin. Conc. H_2SO_4 ⟶ yellow sol. with intense green fluor.
N-*Oxide* : decomp. at 228–30°.

Ullmann, Maag, *Ber.*, 1907, **40**, 2520.
Schmid, Decker, *Ber.*, 1906, **39**, 937.
Kaufmann, Albertini, *Ber.*, 1909, **42**, 2008.
Bernthsen, *Ann.*, 1884, **224**, 13.
Ullmann, *Ann.*, 1907, **355**, 319.
Lehmstedt, Dostal, *Ber.*, 1939, **72**, 804.

4-Phenylacridinic Acid.
See 4-Phenylquinoline-2 : 3-dicarboxylic Acid.

***N*-Phenylacridone**



$C_{19}H_{13}ON$ MW, 271

Yellow cryst. from toluene. M.p. 276°. Sol. boiling C_6H_6, amyl alcohol, toluene with yellow col. Mod. sol. AcOH with intense blue fluor. Spar. sol. MeOH, EtOH. Insol. Et_2O, ligroin. Conc. H_2SO_4 ⟶ yellow sol. with intense green fluor.

Goldberg, Nimcrovsky, *Ber.*, 1907, **40**, 2450.

2-Phenylacrolein.
See Cinnamaldehyde.

1-Phenylacrylic Acid.
See Atropic Acid.

2-Phenylacrylic Acid.
See Cinnamic Acid.

1-Phenyladipic Acid (1-*Phenylbutane*-1 : 4-*dicarboxylic acid*)

C_6H_5·CH·COOH
$[CH_2]_2$
CH_2·COOH

$C_{12}H_{14}O_4$ MW, 222

Cryst. from C_6H_6–Et_2O. M.p. 132–3°.

Case, *J. Am. Chem. Soc.*, 1933, **55**, 2927.
Berlande, *Bull. soc. chim.*, 1942, **9**, 644.

2-Phenyladipic Acid (2-*Phenylbutane*-1 : 4-*dicarboxylic acid*)

CH_2·COOH
C_6H_5·CH
CH_2
CH_2·COOH

$C_{12}H_{14}O_4$ MW, 222

Plates from C_6H_6–Et_2O. M.p. 148°. Sol. EtOH, Me_2CO, Et_2O. Insol. H_2O.
Di-Et ester : $C_{16}H_{21}O_4$. MW, 277. B.p. 197–200°/10 mm.

v. Braun, Weissbach, *Ber.*, 1931, **64**, 1785.
Planske, *J. Am. Chem. Soc.*, 1931, **53**, 1104.

Phenylalanine.
See Aminohydrocinnamic Acid *and* Anilinopropionic Acid.

Phenylaldehydoacetic Acid.
See Phenylformylacetic Acid.

1-Phenylallyl Alcohol (*Vinylphenylcarbinol*, α-*hydroxyallylbenzene*)

C_6H_5·CH(OH)·CH:CH_2

$C_9H_{10}O$ MW, 134

Mobile oil. B.p. 214°/760 mm., 114°/25 mm., 106°/18 mm., 90–2°/11 mm. Decolourises

$KMnO_4$. $O_3 \longrightarrow$ mandelic acid + formic acid. Sweet taste. Odourless.

Et ether: $C_{11}H_{14}O$. MW, 162. Oil. B.p. 203–5°/755 mm. Fruity odour.

Acetoacetyl: b.p. 77°/0·002 mm. D_{25}^{25} 1·0826. n_D^{25} 1·5130.

Benzoyl: mobile oil. B.p. 182°/12 mm.

p-*Nitrobenzoyl*: leaflets from EtOH. M.p. 48°.

Klages, Klenk, *Ber.*, 1906, **39**, 2553.
Rupe, Müller, *Helv. Chim. Acta*, 1921, **4**, 846.
Moureu, Gallagher, *Bull. soc. chim.*, 1921, **29**, 1009.
Meisenheimer, Schmidt, *Ann.*, 1929, **475**, 178.

3-Phenylallyl Alcohol.
See Cinnamyl Alcohol.

Phenylallylamine.
See Cinnamylamine *and under* Aniline.

2-Phenylallylcarbinol (2-*Phenyl*-1-*butenol*-4)

$CH_2{:}C(C_6H_5){\cdot}CH_2{\cdot}CH_2OH$

$C_{10}H_{12}O$ MW, 148

B.p. 123°/10 mm. D^{20} 1·0272. n_D^{20} 1·5577.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1932, **15**, 1266.

3-Phenylallylcarbinol (1-*Phenyl*-1-*butenol*-4, γ-*hydroxy*-α-*butenylbenzene*)

$C_6H_5{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CH_2OH$

$C_{10}H_{12}O$ MW, 148

M.p. 36°. B.p. 99–101°/12 mm. (140°/12 mm.).

Acetyl: b.p. 149°/12 mm. D_{16}^{14} 1·039.

p-*Nitrobenzoyl*: m.p. 119°.

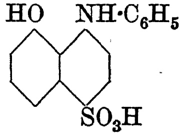
Phenylurethane: m.p. 70°.

Fichter, Kestenholz, *Helv. Chim. Acta*, 1942, **25**, 785.
Normant, *Compt. rend.*, 1948, **226**, 733.

Phenylallylene.
See Methylphenylacetylene.

Phenyl o-aminobenzyl Ether.
See under o-Aminobenzyl Alcohol.

N-Phenyl-1-amino-8-naphthol-4-sulphonic Acid (*Phenyl-S acid*, 8-*anilino*-1-*naphthol*-5-*sulphonic acid*)



$C_{16}H_{13}O_4NS$ MW, 315

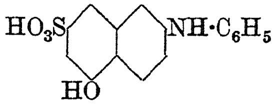
Cryst. Intermediate for azo dyes.

Acid Na salt: needles. Mod. sol. H_2O.

Neutral Na salt: cryst. Mod. sol. cold H_2O.

Bayer, D.R.P., 181,929, (*Chem. Zentr.*, 1907, I, 1653).

N-Phenyl-2-amino-5-naphthol-7-sulphonic Acid (*Phenyl-J acid*, 2-*anilino*-5-*naphthol*-7-*sulphonic acid*, 6-*anilino*-1-*naphthol*-3-*sulphonic acid*)

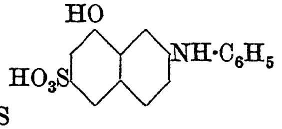


$C_{16}H_{13}O_4NS$ MW, 315

Cryst. Spar. sol. H_2O, EtOH, cold dil. acids. Conc. $H_2SO_4 \longrightarrow$ green sol. Dil. $Na_2CO_3 \longrightarrow$ sol. with violet fluor. Important intermediate for azo dyes.

Leonhardt, D.R.P., 114,248, (*Chem. Zentr.*, 1900, II, 997).
Badische, D.R.P., 122,570, (*Chem. Zentr.*, 1901, II, 670).
Bucherer, Stohmann, *J. prakt. Chem.*, 1905, **71**, 451.

N-Phenyl-2-amino-8-naphthol-6-sulphonic Acid (*Phenyl-gamma acid*, 2-*anilino*-8-*naphthol*-6-*sulphonic acid*, 7-*anilino*-1-*naphthol*-3-*sulphonic acid*)



$C_{16}H_{13}O_4NS$ MW, 315

Needles or leaflets. Sol. 35 parts hot, 300 parts cold H_2O. Intermediate for azo dyes.

Ba salt: leaflets. Mod. sol. H_2O.

Badische, D.R.P. 122,570, (*Chem. Zent.*, 1901, II, 670).
Bucherer, Stohmann, *J. prakt. Chem.*, 1905, **71**, 451.
Cassella, D.R.P., 79,014.
Levinstein, D.R.P., 99,339, (*Chem. Zentr.*, 1899, I, 160).

Phenyl-aminonaphthyl-methane.
See 4-Benzyl-1-naphthylamine.

Phenyl aminotolyl Ketone.
See Amino-methylbenzophenone.

1-Phenyl-n-amyl Alcohol (1-*Phenylpentanol*-1, *butylphenylcarbinol*, α-*hydroxy*-n-*amylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH(C_6H_5)OH$

$C_{11}H_{16}O$ MW, 164

B.p. 137°/21 mm., 132°/14 mm., 121–2°/15 mm.

Urethane: m.p. 75°.

Levene, Mikeska, *J. Biol. Chem.*, 1926, **70**, 355.
Fourneau, Montaigne, Puyal, *Chem. Abstracts*, 1922, **16**, 240.
Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 155.
Ipatieff, Haensel, *J. Am. Chem. Soc.*, 1942, **64**, 520.

3-Phenyl-*n*-amyl Alcohol (3-*Phenyl-pentanol*-1)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_2OH$

$C_{11}H_{16}O$ MW, 164

l-.

B.p. 135–8°/20 mm., 118°/1 mm. $[\alpha]^{25}_{D}$ − 2·69°.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 333.
Lévy, *Compt. rend.*, 1933, **197**, 773.

4-Phenyl-*n*-amyl Alcohol (4-*Phenyl-pentanol*-1)

$CH_3{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_2{\cdot}CH_2OH$

$C_{11}H_{16}O$ MW, 164

l-.

B.p. 109°/1 mm. $[\alpha]^{25}_{D}$ − 0·90°.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 339.

5-Phenyl-*n*-amyl Alcohol (5-*Phenyl-pentanol*-1, ω-*hydroxy*-n-*amylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2OH$

$C_{11}H_{16}O$ MW, 164

B.p. 155°/20 mm., 151°/13 mm.
Phenyl ether: $C_{17}H_{20}O$. MW, 240. Viscous liq. B.p. 198°/14 mm.
Acetyl: b.p. 155°/12 mm.

v. Braun, *Ber.*, 1911, **44**, 2872.
Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 155.

1-Phenyl-*sec.*-*n*-amyl Alcohol.
See Propylbenzylcarbinol.

2-Phenyl-*sec.*-*n*-amyl Alcohol.
See Methylpropylphenylcarbinol.

4-Phenyl-*sec.*-*n*-amyl Alcohol (4-*Phenyl-pentanol*-2)

C_6H_5
$CH_3{\cdot}CH{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_3$

$C_{11}H_{16}O$ MW, 164

B.p. 124–5°/15 mm.

Nenitzescu, Gavat, Cocora, *Ber.*, 1940, **73**, 233.

5-Phenyl-*sec.*-*n*-amyl Alcohol (5-*Phenyl-pentanol*-2, δ-*hydroxy*-n-*amylbenzene*, 1-*phenyl-pentanol*-4)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_3$

$C_{11}H_{16}O$ MW, 164

B.p. 134–5°/16 mm. (140–1°/16 mm.). D^{25}_{4} 0·9643. n^{25}_{5875} 1·51110, n^{19}_{D} 1·518.
Phenylurethane: needles from pet. ether. M.p. 57°.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.
Tiffeneau, Deux, *Compt. rend.*, 1942, **214**, 892.

2-Phenyl-*active*-amyl Alcohol (2-*Methyl-2-phenylbutanol*-1)

CH_3
$CH_3{\cdot}CH_2{\cdot}C{\cdot}CH_2OH$
C_6H_5

$C_{11}H_{16}O$ MW, 164

Oil. B.p. 246°, 138°/23 mm.
Benzoyl: cryst. M.p. 46°. B.p. 202–4°/12 mm.

Blondeau, *Compt. rend.*, 1922, **174**, 1424.

3-Phenyl-*active*-amyl Alcohol (2-*Methyl-3-phenylbutanol*-1)

C_6H_5
$CH_3{\cdot}CH{\cdot}CH{\cdot}CH_2OH$
CH_3

$C_{11}H_{16}O$ MW, 164

Oil with odour of roses. B.p. 132–3°/13 mm.

Braun *et al.*, *Ann.*, 1927, **451**, 48.

2-Phenyl-*n*-amylamine

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2NH_2$

$C_{11}H_{17}N$ MW, 163

l-.

Liq. B.p. 90°/3 mm.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 773.

5-Phenyl-*n*-amylamine (ω-*Amino*-n-*amylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2NH_2$

$C_{11}H_{17}N$ MW, 163

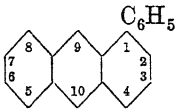
Liq. B.p. 131°/15 mm.
N-*Di-Me*: $C_{13}H_{21}N$. MW, 191. B.p. 134–5°/18 mm.
N-*Et*: $C_{13}H_{21}N$. MW, 191. B.p. 295°. *B,HCl*: m.p. 144°.
N-*Amyl*: $C_{16}H_{27}N$. MW, 233. B.p. 310°. *B,HCl*: cryst. from H_2O or EtOH. M.p. 184–6°.
N-*Cyclohexyl*: $C_{17}H_{28}N$. MW, 246. B.p. 315°. *B,HCl*: m.p. 246°.
Benzoyl: needles from Et_2O–ligroin. M.p. 60°. B.p. 273–5°/15 mm.
B_2,H_2PtCl_6: leaflets from H_2O. M.p. 220°.
Picrate: cryst. from Et_2O. M.p. 152–3°. Sol. EtOH.
Methiodide: leaflets from H_2O. M.p. 181°. Very sol. $CHCl_3$. Spar. sol. H_2O, EtOH.
Methochloroplatinate: light red cryst. from H_2O. M.p. 219°.

Skita, Wulff, *Ann.*, 1927, **455**, 40.
v. Braun, *Ber.*, 1910, **43**, 2849.
Merck, D.R.P., 238,959, (*Chem. Zentr.*, 1911, II, 1284).

Phenylamylene.
See Phenylpentene.

***N*-Phenylanisidine.**
See under 2- and 4-Hydroxydiphenylamines.

1-Phenylanthracene



$C_{20}H_{14}$ MW, 254

Yellow prisms from EtOH. M.p. 110–12°. Sol. usual solvents.

Cook, *J. Chem. Soc.*, 1930, 1091.
Weizmann, Bergmann, Haskelberg, *J. Chem. Soc.*, 1939, 391.

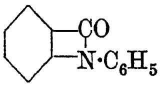
2-Phenylanthracene.
Pale yellow leaflets from AcOEt. M.p. 207–207·5°. Spar. sol. most solvents.

Cook, *J. Chem. Soc.*, 1930, 1091.

9-Phenylanthracene (ms-*Phenylanthracene*).
Leaflets from EtOH. M.p. 152–3°. B.p. 417°. Sol. warm EtOH, Et_2O, $CHCl_3$, CS_2, $CHCl_3$. Sols. show blue fluor.

Baeyer, *Ann.*, 1880, **202**, 61.
Linebarger, *Am. Chem. J.*, 1891, **13**, 554.
Guyot, Catel, *Compt. rend.*, 1905, **140**, 1462.
Bradsher, *J. Am. Chem. Soc.*, 1940, **62**, 486.

***N*-Phenylanthranil**



$C_{13}H_9ON$ MW, 195

Pale yellow needles from 50% EtOH. M.p. 169° decomp. Sol. EtOH, Et_2O, Me_2CO, AcOEt, C_6H_6. Spar. sol. H_2O. Sols. show greenish-blue fluor.

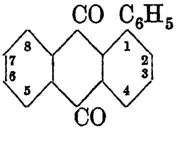
B,HCl: pale yellow needles. M.p. 169° decomp.

B,HgCl₂: golden-yellow leaflets. M.p. 225–32° decomp.

Drechsler, *Monatsh.*, 1914, **35**, 533.

Phenylanthranilic Acid.
See Diphenylamine-2-carboxylic Acid.

1-Phenylanthraquinone



$C_{20}H_{12}O_2$ MW, 284

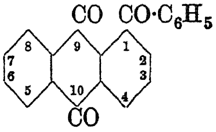
Yellow needles from AcOH. M.p. 176–8°. B.p. 180°/0·2 mm.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 740.
Bergmann, Haskelberg, Bergmann, *J. Org. Chem.*, 1942, **7**, 303.

2-Phenylanthraquinone.
Pale yellow needles from MeOH. M.p. 162–5° (160–1°). Sol. AcOH, CCl_4. Mod. sol. EtOH, Et_2O, $CHCl_3$, Me_2CO, CS_2, C_6H_6. Alc. KOH ⟶ red col. Conc. H_2SO_4 ⟶ red col.

Scholl, Neovius, *Ber.*, 1911, **44**, 1079.
Groggins, *Ind. Eng. Chem.*, 1930, **22**, 620.

Phenyl 1-anthraquinonyl Ketone (1-*Benzoylanthraquinone*)



$C_{21}H_{12}O_3$ MW, 312

Yellowish cryst. powder from AcOH. M.p. 229° (225–7°). Sol. conc. H_2SO_4 with pale yellow col.

1-*Oxime*: yellow rhombic leaflets. Decomp. about 218°.

9-*Oxime*: *syn.*, yellow rhombic cryst. Decomp. about 210°. *Anti.*: yellow needles or leaflets. M.p. 230°.

Scholl, Müller, Stix, *Ber.*, 1935, **68**, 801.
Scholl *et al.*, *Ann.*, 1934, **512**, 1.
I.G., D.R.P., 487,254, (*Chem. Abstracts*, 1930, **24**, 2147).
Schaarschmidt, *Ber.*, 1915, **48**, 837.

Phenylarsen-dibromide.
See Phenyldibromoarsine.

Phenylarsen-dichloride.
See Phenyldichloroarsine.

Phenylarsine (*Arsinobenzene*)

$C_6H_5{\cdot}AsH_2$

C_6H_7As MW, 154

Oil. B.p. 148°, 93°/70 mm., 77°/33 mm., 55°/14 mm., 36°/2 mm. Sol. EtOH, Et_2O. In air ⟶ phenylarsinic acid.

As-*Di-Me*: $C_8H_{11}As$. MW, 182. Liq. B.p. 200°. Sol. EtOH, C_6H_6. Insol. H_2O. *Methiodide*: prisms from EtOH. M.p. 248·5° (244°). *Methochloroplatinate*: red plates from hot H_2O. M.p. 219° decomp.

As-*Di-Et*: $C_{10}H_{15}As$. MW, 210. B.p. 240°. *Methiodide*: prisms from EtOH–Et_2O. M.p. 122° (77°). *Ethiodide*: prisms from H_2O. M.p. 112–13°. Sol. EtOH. Insol. Et_2O.

As-*Et-Propyl*: $C_{11}H_{17}As$. MW, 224. B.p. 245°.

As-*Phenyl*: *see* Diphenylarsine.

As-*Diphenyl*: *see* Triphenylarsine.

Palmer, Dehn, *Ber.*, 1901, **34**, 3598.
Fichter, Elkind, *Ber.*, 1916, **49**, 245.
Winmill, *J. Chem. Soc.*, 1912, **101**, 719.
Michaelis, Link, *Ann.*, 1881, **201**, 205.
La Coste, Michaelis, *Ann.*, 1880, **201**, 212.

Phenylarsinic Acid (*Phenylarsonic acid*)

$C_6H_5{\cdot}As(OH)_2$
$\ddot{O}$

$C_6H_7O_3As$ MW, 202

Cryst. from H_2O. M.p. 158–62° decomp. Above m.p. ⟶ anhydride. 3·25 parts sol. 100 parts H_2O at 28°, 24 parts at 84°. Very stable to ox. agents.

Anhydride: amorph. Decomp. on heating.

Di-Me ester: $C_8H_{11}O_3As$. MW, 230. B.p. 188°/95 mm. D^{23} 1·3946. Decomp. by H_2O.

Di-Et ester: $C_{10}H_{15}O_3As$. MW, 258. B.p. 168–70°/15 mm. D^{15} 1·318.

Dichloride: $C_6H_5OCl_2As$. MW, 239. Cryst. M.p. about 100°. Sol. H_2O with decomp.

La Coste, Michaelis, *Ann.*, 1880, **201**, 203.
Michaelis, Loesner, *Ber.*, 1884, **27**, 265.
Michaelis, *Ann.*, 1902, **320**, 293.
Bullard, Dickey, *Organic Syntheses*, 1935, XV, 59.

Phenylarsonic Acid.
See Phenylarsinic Acid.

N-Phenylaspartic anil.
See Anilinosuccinanil.

Phenylaticonic Acid (*Iso-γ-phenylitaconic acid*)

$$\begin{matrix} C_6H_5{\cdot}CH \\ \| \\ HOOC{\cdot}C{\cdot}CH_2{\cdot}COOH \end{matrix}$$

$C_{11}H_{10}O_4$ MW, 206

Prisms from Et_2O. M.p. 149–51° (not sharp). Sol. 15 parts Et_2O, 90 parts H_2O at ord. temps. Spar. sol. $CHCl_3$. Insol. CS_2, ligroin. Sunlight ⟶ phenylitaconic acid. Heat in vacuo at 160–75° ⟶ anhydride. $KMnO_4$ ⟶ benzaldehyde + malonic acid + oxalic acid.

Anhydride: needles from Et_2O–$CHCl_3$. M.p. 138–40°. Sol. $CHCl_3$, C_6H_6. Spar. sol. Et_2O.

Stobbe, Horn, *Ber.*, 1908, **41**, 3983.
Stobbe, *Ber.*, 1908, **41**, 4353.
Fittig, Brooke, *Ann.*, 1899, **305**, 33.

Phenyl azide (*Azidobenzene, diazobenzolimide, phenylazoimide, triazobenzene*)

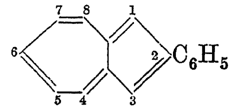
$$C_6H_5{\cdot}N{<}\begin{matrix} N \\ \| \\ N \end{matrix} \quad \text{or} \quad C_6H_5{\cdot}N{:}N{:}N$$

$C_6H_5N_3$ MW, 119

Pale yellow oil. B.p. 73·5°/22–4 mm., 59°/14 mm. Mod. sol. EtOH, Et_2O. Insol. H_2O. D_0^4 1·12399, D^{10} 1·098, D^{25} 1·0853. n_D^{11} 1·56104. Possesses aromatic ammoniacal odour. Explodes on dist. at ord. press., or by treatment with conc. H_2SO_4. Volatile in steam. Strongly antipyretic. $N_2H_4{\cdot}H_2O$ ⟶ $C_6H_6 + NH_3 + N$. NaHg + EtOH ⟶ hydrazobenzene. Zn + H_2SO_4 ⟶ $C_6H_6 + NH_3 + N$. Dil. H_2SO_4 ⟶ *p*-aminophenol. Na + EtOH ⟶ aniline + N. Long heating with conc. HCl ⟶ *o*- and *p*-chloroanilines.

Dimroth, *Ber.*, 1902, **35**, 1032 (*Note*).
Lindsay, Allen, *Organic Syntheses*, 1942, XXII, 96.

2-Phenylazulene



$C_{16}H_{12}$ MW, 204

Blue cryst. from C_6H_6. M.p. 230°.

Plattner, Sandrin, Wyss, *Helv. Chim. Acta*, 1946, **29**, 1604.

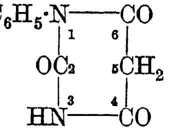
4-Phenylazulene.
Blue oil.

Picrate: black needles from EtOH. M.p. 80–1°.

sym.-*Trinitrobenzene add. comp.*: black needles from EtOH. M.p. 86–7°.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1936, **19**, 878.

1-Phenylbarbituric Acid



$C_{10}H_8O_3N_2$ MW, 204

Cryst. from EtOH. M.p. 262°.

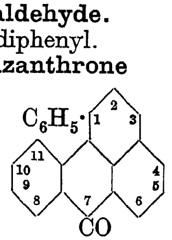
Macbeth, Nunan, Traill, *J. Chem. Soc.*, 1926, 1252.
Winslow, *J. Am. Chem. Soc.*, 1939, **61**, 2089.
Aspelund, Lindh, *Chem. Abstracts*, 1939, **33**, 6802; 1943, **37**, 5028.

5-Phenylbarbituric Acid.
M.p. 258° (250°, 206°).

Lund, *Chem. Zentr.*, 1936, I, 2096.
Bayer, D.R.P., 249,722, (*Chem. Zentr.*, 1912, II, 652).
Aspelund, *Chem. Abstracts*, 1939, **33**, 8182.
Aspelund, Lindh, *Chem. Abstracts*, 1943, **37**, 5028.

Phenylbenzaldehyde.
See Aldehydodiphenyl.

1-Phenylbenzanthrone



$C_{23}H_{14}O$ MW, 306

M.p. 186°. Sol. conc. H_2SO_4 with orange-red col. showing strong fluor. $Na_2Cr_2O_7$ ⟶ anthraquinone-1-carboxylic acid.

Clar, *Ber.*, 1932, **65**, 858.
I.G., B.P. 286,685, (*Chem. Abstracts*, 1929, **23**, 1139).
Nakanishi, *Chem. Abstracts*, 1934, **28**, 763.

2-Phenylbenzanthrone.

Yellow needles from AcOH. M.p. 199–200°. Sol. H_2SO_4 to red sol. changing to orange-red.

I.G., B.P. 297,129, (*Chem. Abstracts*, 1929, **23**, 2580).

3-Phenylbenzanthrone.

Yellow needles from AcOH. M.p. 182–3° (181°). Ox. ⟶ phenyl 1-anthraquinonyl ketone.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 1323.
Nakanishi, *Chem. Abstracts*, 1934, **28**, 763.
I.G., D.R.P., 488,607, (*Chem. Abstracts*, 1930, **24**, 2148); B.P. 297,129, (*Chem. Abstracts*, 1929, **23**, 2580).

6-Phenylbenzanthrone.

M.p. 186°. Alk. $KMnO_4$ ⟶ 4-phenylanthraquinone-1-carboxylic acid.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 743.

10-Phenylbenzanthrone.

Golden-yellow or yellowish-brown leaflets from C_6H_6 or Py.Aq. M.p. 178–9° (170–1°). Very sol. C_6H_6, CS_2, Py. Mod. sol. AcOH. Spar. sol. EtOH. Very spar. sol. pet. ether. Sol. conc. H_2SO_4 with red col. showing golden-yellow fluor.

Scholl, Seer, *Ann.*, 1912, **394**, 150.
Schmidlin, Garcia-Banùs, *Ber.*, 1912, **45**, 3184.

Phenylbenzene.

See Diphenyl.

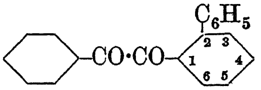
Phenylbenzhydrylcarbinol.

See 2-Hydroxy-1 : 1 : 2-triphenylethane.

Phenyl benzhydryl Ketone.

See Diphenylbenzoylmethane.

2-Phenylbenzil



$C_{20}H_{14}O_2$ MW, 286

Yellow plates from EtOH. M.p. 80°. Ox. ⟶ diphenyl-2-carboxylic acid.

Quinoxaline deriv.: m.p. 163°.

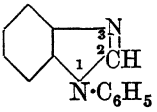
Kohler, Nygaard, *J. Am. Chem. Soc.*, 1930, **52**, 4137.

4-Phenylbenzil.

Yellow plates from EtOH. M.p. 105°.

Hatt, Pilgrim, Hurran, *J. Chem. Soc.*, 1936, 94.
Gomberg, Natta, *J. Am. Chem. Soc.*, 1929, **51**, 2244.

1-Phenylbenziminazole



$C_{13}H_{10}N_2$ MW, 194

Needles from ligroin. M.p. 98°. Sol. MeOH, EtOH. Spar. sol. H_2O.

B,HCl,HgCl₂: cryst. from EtOH.Aq. M.p. 145–146·5°.

Fischer, Rigaud, *Ber.*, 1901, **34**, 4204.
Phillips, *J. Chem. Soc.*, 1929, 2823.

2-Phenylbenziminazole.

Plates from AcOH, needles from H_2O. M.p. 291° (280°, 285°). Sol. AcOH. Mod. sol. EtOH. Spar. sol. H_2O, $CHCl_3$, C_6H_6. Forms metallic derivs. with silver and mercury.

B,HCl: needles from EtOH. M.p. 303° (above 306°).

Feigl, Gleich, *Monatsh.*, 1928, **49**, 394.
Fischer, Mann-Tiechler, *J. prakt. Chem.*, 1924, **107**, 45.
Guha, Ray, *J. Indian Chem. Soc.*, 1925, **2**, 93.
Weiderhagen, *Ber.*, 1936, **69**, 2263.
Hölljes, Wagner, *J. Org. Chem.*, 1944, **9**, 31.
Poraĭ-Koshits *et al.*, *J. Gen. Chem. U.S.S.R.*, 1947, **17**, 1768, (*Chem. Abstracts*, 1948, **42**, 5903).

Phenyl benzoate

$C_6H_5·CO·OC_6H_5$

$C_{13}H_{10}O_2$ MW, 198

Prisms. M.p. 71°. B.p. 314° (299°). Sol. hot EtOH, hot Et_2O. Insol. H_2O. Heat of comb. C_p 1511·3 Cal.

Rasinski, *J. prakt. Chem.*, 1882, **26**, 62.
Reychler, *Chem. Zentr.*, 1908, I, 1042.
Czapek, *Monatsh.*, 1914, **35**, 637.

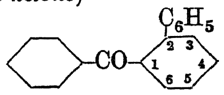
Phenylbenzoic Acid.

See Diphenyl-carboxylic Acid.

α-Phenylbenzoin.

See Diphenylbenzoylcarbinol.

2-Phenylbenzophenone (*2-Benzoyldiphenyl, phenyl* o-*xenyl ketone*)



$C_{19}H_{14}O$ MW, 258

Cryst. from ligroin. M.p. 90°.

Anil: m.p. 91–2°.

Hatt, Pilgrim, Stephenson, *J. Chem. Soc.*, 1941, 478.
Bradsher, *J. Am. Chem. Soc.*, 1944, **66**, 45.

3-Phenylbenzophenone (*3-Benzoyldiphenyl, phenyl* m-*xenyl ketone*).

Colourless cryst. from pet. ether. M.p. 79°. B.p. 264–7°/25 mm.

Hatt, Pilgrim, Stephenson, *J. Chem. Soc.*, 1941, 478.

4-Phenylbenzophenone (*4-Benzoyldiphenyl, phenyl* p-*xenyl ketone*).

Cryst. from EtOH. M.p. 102° (106°). B.p. 419–20°/744 mm. Sol. Et_2O, $CHCl_3$, C_6H_6, hot EtOH. $CrO_3 \longrightarrow$ *p*-benzoylbenzoic acid.

Oxime: needles from EtOH. M.p. 193–4°. Sol. EtOH, Et_2O.

Nenitzescu, Isacescu, Ionescu, *Ann.*, 1931, **491**, 217.
Hey, Jackson, *J. Chem. Soc.*, 1936, 805.
Long, Henze, *J. Am. Chem. Soc.*, 1941, **63**, 1939.

Phenylbenzoylacetic Acid (*2-Keto-1 : 2-diphenylpropionic acid*)

$$C_6H_5{\cdot}CO{\cdot}\underset{}{CH}(C_6H_5){\cdot}COOH$$

$C_{15}H_{12}O_3$ MW, 240

Free acid unknown.

Me ester: $C_{16}H_{14}O_3$. MW, 254. Liq. Decomp. on dist. $\longrightarrow$ stilbene.

Et ester: $C_{17}H_{16}O_3$. MW, 268. Prisms. M.p. 90°. Very sol. EtOH, Et_2O, C_6H_6.

l-*Menthyl ester*: (i) *keto form*, needles from MeOH. M.p. 116°. Sol. Et_2O, C_6H_6. Spar. sol. cold EtOH. $[\alpha]_D^{20} + 21{\cdot}10°$ in C_6H_6, $- 12{\cdot}1°$ in EtOH. Et_2O sol. + $FeCl_3 \longrightarrow$ deep red col. (ii) *Enol form*: $[\alpha]_D^{20} - 62{\cdot}83°$ in C_6H_6, $- 62{\cdot}6°$ in EtOH.

Amide: $C_{15}H_{13}O_2N$. MW, 239. Needles from EtOH. M.p. 178°. Sol. EtOH. Spar. sol. Et_2O, hot H_2O.

Nitrile: $C_{15}H_{11}ON$. MW, 221. Plates from AcOH. M.p. 93–4°. Very sol. EtOH, Et_2O, C_6H_6, hot pet. ether. Alk. hyd. $\longrightarrow$ benzoic acid + benzyl cyanide. Conc. H_2SO_4 at 120° $\longrightarrow$ amide. Conc. HCl at 150° $\longrightarrow$ deoxybenzoin. Boiling dil. acids $\longrightarrow$ benzoic + phenylacetic acids.

Anilide: needles from EtOH. M.p. 168–9°.

Oxime: cryst. M.p. 138–9°.

Rupe, Gisiger, *Helv. Chim. Acta*, 1925, **8**, 349.
Ghosh, *J. Chem. Soc.*, 1916, **109**, 117.
Walther, Schickler, *J. prakt. Chem.*, 1897, **55**, 314.
Shivers, Dillon, Hauser, *J. Am. Chem. Soc.*, 1947, **69**, 119.

3-Phenyl-1-benzoylbutyric Acid

$$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH(CO{\cdot}C_6H_5){\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Free acid not isolated.

Et ester: $C_{19}H_{20}O_3$. MW, 296. B.p. 225–30°/11 mm.

Auwers, Möller, *J. prakt. Chem.*, 1925, **109**, 150.

1-Phenyl-2-benzoylbutyric Acid

$$CH_3{\cdot}CH(C_6H_5{\cdot}CO){\cdot}CH(C_6H_5){\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Exists in two forms. (i) Needles from $CHCl_3$–ligroin. M.p. 145°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. ligroin. (ii) Needles from $CHCl_3$–ligroin. M.p. 131°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. ligroin, cold H_2O.

Me ester: $C_{18}H_{18}O_3$. MW, 282. Exists in two forms. (i) Plates from MeOH. M.p. 105°. (ii) M.p. 87°.

Reimer, Reynolds, *Am. Chem. J.*, 1912, **48**, 216, 218.

3-Phenyl-2-benzoylbutyric Acid

$$C_6H_5{\cdot}CH_2{\cdot}CH(CO{\cdot}C_6H_5){\cdot}CH_2{\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Needles from C_6H_6–ligroin. M.p. 100–1°.

Borsche, *Ber.*, 1914, **47**, 1114.

2-Phenyl-3-benzoylbutyric Acid (*β-Phenacylhydrocinnamic acid*)

$$C_6H_5{\cdot}CO{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Needles or plates from H_2O, prisms from AcOH. M.p. 160° (152–153·5°). Sol. EtOH, AcOH. Spar. sol. Et_2O. Hot $Ac_2O \longrightarrow$ lactone.

Me ester: $C_{18}H_{18}O_3$. MW, 282. Needles. M.p. 94°.

Et ester: $C_{19}H_{20}O_3$. MW, 296. M.p. 59–61°.

Lactone: $C_{17}H_{14}O_2$. MW, 250. Needles. Sol. Et_2O. Hot alc. NaOH regenerates the acid.

Oxime: m.p. 144–145·5°.

Semicarbazone: cryst. from 90% EtOH. M.p. 220° (212·5–213°).

Stobbe, *Ber.*, 1901, **34**, 655.
Vorländer, Knötzsch, *Ann.*, 1897, **294**, 332.
Kohler, Steele, *J. Am. Chem. Soc.*, 1919, **41**, 1103.
Qudrat-i-Khuda, *J. Indian Chem. Soc.*, 1931, **8**, 220.
Breslow, Hauser, *J. Am. Chem. Soc.*, 1940, **62**, 2385.

3-Phenyl-3-benzoylbutyric Acid (*2-Desylpropionic acid*)

$$C_6H_5{\cdot}CO{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_2{\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Needles from EtOH. M.p. 136° (133–4°). Sol. EtOH, AcOH. Insol. H_2O.

Me ester: needles. M.p. 63–4°.

Et ester: needles. M.p. 33–4°.

Knoevenagel, *Ber.*, 1888, **21**, 1351.
Meerwein, *J. prakt. Chem.*, 1918, **97**, 261.

Phenylbenzoyldiazomethane.
See Azibenzil.

2-Phenyl-1-benzoylisobutyric Acid (*Methylbenzylbenzoylacetic acid, 1-benzyl-1-benzoylpropionic acid*)

$$\begin{matrix} CH_3 \\ C_6H_5{\cdot}CH_2 \end{matrix} > C < \begin{matrix} CO{\cdot}C_6H_5 \\ COOH \end{matrix}$$

$C_{17}H_{16}O_3$ MW, 268

Free acid not isolated.
Et ester: $C_{19}H_{20}O_3$. MW, 296. Oil. B.p. 223–8°/19 mm.

Hope, Perkin, *J. Chem. Soc.*, 1909, **95**, 2046.

2-Phenyl-2-benzoylisobutyric Acid (1-*Desylpropionic acid*)

$$\begin{matrix} C_6H_5 \\ C_6H_5{\cdot}CO \end{matrix} > CH{\cdot}CH < \begin{matrix} CH_3 \\ COOH \end{matrix}$$

$C_{17}H_{16}O_3$ MW, 268

Needles from EtOH. M.p. 220° (213·5°). Insol. H_2O.

Knoevenagel, *Ber.*, 1888, **21**, 1353.
Japp, Michie, *J. Chem. Soc.*, 1903, **83**, 299.

2-Phenyl-2′-benzoylisobutyric Acid (*α-Phenacylhydrocinnamic acid, 1-benzyl-2-benzoylpropionic acid*)

$$\begin{matrix} C_6H_5{\cdot}CO{\cdot}CH_2 \\ C_6H_5{\cdot}CH_2 \end{matrix} > CH{\cdot}COOH$$

$C_{17}H_{16}O_3$ MW, 268

Needles from MeOH. M.p. 176° (169·5°). Sol. EtOH, $CHCl_3$, AcOH. Mod. sol. MeOH. Spar. sol. Et_2O. Insol. H_2O, pet. ether. Sublimes.
Me ester: $C_{18}H_{18}O_3$. MW, 282. Prisms from MeOH. M.p. 68·5°.

Thiele, Mayr, *Ann.*, 1899, **306**, 186.
Klobb, *Bull. soc. chim.*, 1897, **17**, 411.
Kohler, *Ber.*, 1905, **38**, 1206.

2-Phenyl-1-benzoylpropionic Acid (*Benzylbenzoylacetic acid, α-benzoylhydrocinnamic acid*)

$$\begin{matrix} C_6H_5{\cdot}CO \\ C_6H_5{\cdot}CH_2 \end{matrix} > CH{\cdot}COOH$$

$C_{16}H_{14}O_3$ MW, 254

Me ester: $C_{17}H_{16}O_3$. MW, 268. B.p. 250–55°/50 mm.
Et ester: $C_{18}H_{18}O_3$. MW, 282. B.p. 265–70°/80 mm., 218–20°/12 mm.
l-*Menthyl ester*: needles. M.p. 117°. $[\alpha]_D^{20}$ — 60·83° in C_6H_6.

Perkin, Calman, *J. Chem. Soc.*, 1886, **49**, 155.
Perkin, Stenhouse, *J. Chem. Soc.*, 1891, **59**, 1006.
Jacobson, Ghosh, *J. Chem. Soc.*, 1915, **107**, 961.

1-Phenyl-2-benzoylpropionic Acid (*Phenylphenacylacetic acid*)

$$C_6H_5{\cdot}CO{\cdot}CH_2{\cdot}\overset{C_6H_5}{CH}{\cdot}COOH$$

$C_{16}H_{14}O_3$ MW, 254

d-.
Plates from Et_2O. M.p. 176–8°. $[\alpha]_D$ + 157·3° in AcOEt.
Semicarbazone: needles from MeOH. M.p. 107–10°, resolidifying on further heating, melting again above 160°.

l-.
Plates from Et_2O. M.p. 176–8°. $[\alpha]_D^{16}$ — 157·6° in AcOEt.

dl-.
Prisms from EtOH. M.p. 153° (160°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. hot H_2O.
Me ester: cryst. from MeOH. M.p. 104°.
Et ester: needles. M.p. 37–8°.
Amide: $C_{16}H_{15}O_2N$. MW, 253. Needles from EtOH. M.p. 149°.
Nitrile: $C_{16}H_{13}ON$. MW, 235. Plates from EtOH. M.p. 127·5°. Sol. EtOH, Et_2O, $CHCl_3$.
Oxime: plates + 1 mol. C_6H_6 from C_6H_6. M.p. 83–7°.
Semicarbazone: cryst. powder from EtOH. M.p. 189–91°.

Lapworth, Wechsler, *J. Chem. Soc.*, 1910, **97**, 41.
Allen, Kimball, *Organic Syntheses*, 1930, X, 80.
Hann, Lapworth, *J. Chem. Soc.*, 1904, **85**, 1368.
Rupe, Schneider, *Ber.*, 1895, **28**, 962.

2-Phenyl-2-benzoylpropionic Acid (*β-Benzoylhydrocinnamic acid, desylacetic acid*)

$$C_6H_5{\cdot}CO{\cdot}\overset{C_6H_5}{CH}{\cdot}CH_2{\cdot}COOH$$

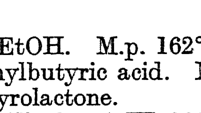
$C_{16}H_{14}O_3$ MW, 254

Cryst. from EtOH. M.p. 162°. Fuming HI → 2 : 3-diphenylbutyric acid. NaHg → 2 : 3-diphenyl-3-butyrolactone.
Me ester: $C_{17}H_{16}O_3$. MW, 268. Cryst. from MeOH. M.p. 49°.

Thiele, Straus, *Ann.*, 1901, **319**, 164.
Anschütz, Walter, *Ann.*, 1907, **354**, 147.

2-Phenyl-γ-benzpyrone.
See Flavone.

2-Phenylbenzthiazole



$C_{13}H_9NS$ MW, 211

Needles from EtOH.Aq. M.p. 114°.
Methiodide: m.p. 218°.

Methopicrate: orange-yellow plates. M.p. 125–6°.
Methoperchlorate: needles. M.p. 220°.
Methochloroplatinate: m.p. 243° decomp.
Dibromide: m.p. 119°.
Tetrabromide: m.p. 134–6°.
Hexabromide: m.p. 156° decomp.
$C_{13}H_9NS,C_6H_3(NO_2)_3$-1 : 3 : 5 : yellow needles. M.p. 106°.

Chandri, Desai, Hunter, *J. Indian Chem. Soc.*, 1934, **11**, 252.
Hunter, *J. Chem. Soc.*, 1930, 138.
Clark, *J. Chem. Soc.*, 1928, 2316.
Chapman, *ibid.*, 1894.
Bogert, Abrahamson, *J. Am. Chem. Soc.*, 1922, **44**, 831.
Gilman, Beel, *J. Am. Chem. Soc.*, 1949, **71**, 2328.

Phenylbenzylacetic Acid.
See 1 : 2-Diphenylpropionic Acid.
Phenylbenzyl Alcohol.
See Hydroxymethyl-diphenyl.
Phenylbenzylamine.
See Benzylaniline.
Phenylbenzylcarbinol (1 : 2-*Diphenylethyl alcohol*, α-*hydroxydiphenylethane*, α-*hydroxydibenzyl*)

$C_6H_5 \cdot CH(OH) \cdot CH_2 \cdot C_6H_5$

$C_{14}H_{14}O$ MW, 198

dl-.
Needles from pet. ether–benzene. M.p. 67°. B.p. 177°/15 mm.
Acetyl: b.p. 202–5°/10 mm.
Benzoyl: needles. M.p. 70°.
Hydrogen phthaloyl: needles from pet. ether–Et_2O. M.p. 131°.
Et ether: α-ethoxydibenzyl. $C_{16}H_{18}O$. MW, 226. B.p. 149–51°/10 mm. Hot dil. H_2SO_4 ⟶ stilbene.
Phenylurethane: m.p. 94–5°.

d-.
Needles from Et_2O–pet. ether. M.p. 67°. D_4^{70} 1·0358. $[\alpha]_D^{25}$ − 8·5° in $CHCl_3$, + 53° in EtOH.
Hydrogen phthaloyl: needles from Et_2O–pet. ether. M.p. 131°. $[\alpha]_D^{25}$ + 33° in EtOH. *Quinine salt*: needles from EtOH. M.p. 205°.

l-.
Needles from Et_2O–pet. ether. M.p. 67°.
Acetyl: b.p. 182°/15 mm. D_4^{17} 1·0831. $[\alpha]_D^{17}$ + 23·5°.
Hydrogen phthaloyl: needles from Et_2O–pet. ether. M.p. 131°. $[\alpha]_{5461}^{25}$ − 39° in EtOH. *Cinchonine salt*: cryst. from MeOH–Me_2CO. M.p. 110°.

Gerrard, Kenyon, *J. Chem. Soc.*, 1928, 2564.
Tuat, Guyard, *Bull. soc. chim.*, 1947, 1086.

Phenyl benzyl Diketone (*Benzoylphenacetyl*, *phenylbenzylglyoxal*, 1 : 3-*diphenylpropandione*-1 : 2, 1 : 2-*diketo*-1 : 3-*diphenylpropane*)

$C_6H_5 \cdot \underset{\beta}{C}O \cdot \underset{\alpha}{C}O \cdot CH_2 \cdot C_6H_5$

or

$C_6H_5 \cdot CO \cdot C(OH) \vdots CH \cdot C_6H_5$

$C_{15}H_{12}O_2$ MW, 224

Three modifications have been described:

(i) Yellow needles. M.p. 67°.
(ii) Yellowish-white cryst. M.p. 90°.
(iii) Lemon-yellow needles. M.p. 35–6°. B.p. 191–2°/15 mm.

(i) and (ii) are probably stereoisomers of the enolic form. (iii) is the ketonic form.
Readily oxidised in air.
α-*Oxime*: ω-isonitroso-ω-benzylacetophenone. Plates from C_6H_6. M.p. 126°. Sol. alkalis with yellow col.
Dioxime: phenylbenzylglyoxime. Plates from AcOH. M.p. 207° (217–18°). Sol. alkalis, reppd. by acids.
Di-phenylhydrazone: m.p. 131°.

Wieland, *Ber.*, 1903, **36**, 3018.
Widman, *Ber.*, 1916, **49**, 484.
Jorländer, *Ber.*, 1917, **50**, 416.
Moureu, *Ann. chim.*, 1930, **14**, 301.
Kohler, Barnes, *J. Am. Chem. Soc.*, 1934, **56**, 211.

Phenyl benzyl Ether

$C_6H_5 \cdot CH_2 \cdot O \cdot C_6H_5$

$C_{13}H_{12}O$ MW, 184

Leaflets from EtOH. M.p. 40°. B.p. 286–7°, 178–9°/35 mm., 124–5°/4 mm. Conc. HCl at 100° ⟶ phenol + benzyl chloride.

van Duzee, Adkins, *J. Am. Chem. Soc.*, 1935, **57**, 148.
Baw, *J. Indian Chem. Soc.*, 1926, **3**, 103.
Powell, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 656.

***sym.*-Phenylbenzylethylene.**
See 1 : 3-Diphenylpropylene.
Phenylbenzylglycollic Acid.
See 1 : 2-Diphenyl-lactic Acid.
Phenylbenzylglyoxal.
See Phenyl benzyl Diketone.
Phenylbenzylglyoxime.
See under Phenyl benzyl Diketone.
***sym.*-Phenylbenzylhydrazine** (β-*Benzylphenylhydrazine*)

$C_6H_5 \cdot CH_2 \cdot NH \cdot NH \cdot C_6H_5$

$C_{13}H_{14}N_2$ MW, 198

Colourless cryst. M.p. 35°. B.p. about 290° decomp. Very sol. prac. all org. solvents. Oxidises in air to benzaldehyde phenylhydrazone.

B,HCl: leaflets from H_2O. M.p. 205° (193–5°). Spar. sol. EtOH, cold H_2O. Insol. Et_2O.
B,(COOH)$_2$: m.p. 190°.
N-*Acetyl*: m.p. 92°.

Ponzio, Valente, *Gazz. chim. ital.*, 1908, 38, i, 525.
Schlenk, *J. prakt. Chem.*, 1908, **78**, 51.
Grammaticakis, *Compt. rend.*, 1937, **204**, 1262.
Gilman, Adams, Pratt, *J. Org. Chem.*, 1938–39, **3**, 99.

***unsym.*-Phenylbenzylhydrazine** (*α-Benzylphenylhydrazine*)

$$\begin{matrix} C_6H_5 \\ C_6H_5{\cdot}CH_2 \end{matrix}\!\!>N{\cdot}NH_2$$

$C_{13}H_{14}N_2$ MW, 198

Colourless oil. B.p. 216–18°/38 mm., 207–8°/10 mm., 157–9°/4 mm. Part. decomp. on standing.
B,HCl: needles from H_2O. M.p. 170° (166–7°).
d-*Camphor-β-sulphonic acid salt*: prisms from AcOEt–Et_2O. M.p. 156–7°.
Methochloride: prisms. M.p. 158–9° decomp. Very sol. H_2O, EtOH. Insol. Et_2O, C_6H_6. *B,AuCl*$_3$: yellow prisms from dil. HCl. M.p. 134° decomp. *B*$_2$*,PtCl*$_4$: orange needles from dil. HCl. M.p. 162° decomp.
Methiodide: cryst. from EtOH. M.p. 122° decomp. *B*$_2$*,HgI*$_2$: prisms from MeOH. M.p. 135–6° decomp. *B,HgI*$_2$: yellow prisms from EtOH. M.p. 128–9° decomp.
N'-*Acetyl*: m.p. 120–1°.

Ofner, *Monatsh.*, 1904, **25**, 599.
Milrath, *Monatsh.*, 1908, **29**, 910 (*Footnote* 2).
Ruff, Ollendorff, *Ber.*, 1899, **32**, 3235 (*Footnote* 2).
Ponzio, Valente, *Gazz. chim. ital.*, 1908, **38**, i, 520.
Michaelis, Philips, *Ann.*, 1889, **252**, 286.
Singh, *J. Chem. Soc.*, 1914, **105**, 1976.
Audrieth, Weisiger, Carter, *J. Org. Chem.*, 1941, **6**, 418.

Phenylbenzylideneacetone.
See Benzyl styryl Ketone.
Phenylbenzylidenebutyric Acid.
See Diphenylbutylene-carboxylic Acid.
Phenylbenzylidenelactic Acid.
See 1-Hydroxy-2 : 3-diphenylvinylacetic Acid.
Phenyl benzyl Ketone.
See Deoxybenzoin.
Phenylbenzylnitrosamine.
See under Benzylaniline.
Phenylbenzylsuccinic Acid.
See 1 : 3-Diphenylpropane-1 : 2-dicarboxylic Acid.
Phenyl benzyl sulphide.
See under Benzyl Mercaptan.

Phenyl benzyl sulphone

$$C_6H_5{\cdot}SO_2{\cdot}CH_2{\cdot}C_6H_5$$

$C_{13}H_{12}O_2S$ MW, 232

Cryst. from EtOH. M.p. 146–146·5°.

Shriner, Struck, Jorison, *J. Am. Chem. Soc.*, 1930, **52**, 2060.

***sym.*-Phenylbenzylurea**

$$\underset{4}{C_6H_5{\cdot}CH_2}{\cdot}\underset{3}{NH}{\cdot}\underset{2}{CO}{\cdot}\underset{1}{NH}{\cdot}C_6H_5$$

$C_{14}H_{14}ON_2$ MW, 226

Needles from EtOH. M.p. 170° (168°). Sol. EtOH. Insol. H_2O.
1-N-*Me*: $C_{15}H_{16}ON_2$. MW, 240. M.p. 84°. Sol. EtOH, AcOH, C_6H_6.

Ley, Krafft, *Ber.*, 1907, **40**, 703.
Thiele, Pickard, *Ann.*, 1899, **309**, 203.
Kühn, Riesenfeld, *Ber.*, 1891, **24**, 3817.

Phenylbenzylvinylacetic Acid.
See 1 : 4-Diphenyl-2-butylene-1-carboxylic Acid.

ω-Phenylbiuret (*Allophanic acid anilide, 1-phenylbiuret, allophanylaniline*)

$$C_6H_5{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}CO{\cdot}NH_2$$

$C_8H_9O_2N_3$ MW, 179

Cryst. from EtOH.Aq. M.p. 167° (various m.p.'s between 156–65° are recorded, due to decomp. on melting). Sol. EtOH. Insol. Et_2O. Does not give biuret reaction. Heat $\longrightarrow$ NH_3 + cyanuric acid + carbanilide. Heat with aniline $\longrightarrow$ carbanilide.

Davis, Blanchard, *J. Am. Chem. Soc.*, 1929, **51**, 1809.
Dains, Wertheim, *J. Am. Chem. Soc.*, 1920, **42**, 2307.
Gatewood, *J. Am. Chem. Soc.*, 1925, **47**, 410.
Blair, *J. Am. Chem. Soc.*, 1934, **56**, 904.

***ms.*-Phenylbiuret** (*3-Phenylbiuret, dicarbamylaniline*)

$$C_6H_5{\cdot}N{<}\begin{matrix} CONH_2 \\ CONH_2 \end{matrix}$$

$C_8H_9O_2N_3$ MW, 179

Needles or prisms from hot H_2O. M.p. 192°. Very sol. EtOH, boiling H_2O. Heat with aniline $\longrightarrow$ carbanilide. KOH + a little $CuSO_4$ $\longrightarrow$ reddish-violet col.

Blair, *J. Am. Chem. Soc.*, 1934, **56**, 904.
Schiff, *Ann.*, 1907, **352**, 78.
Weith, *Ber.*, 1877, **10**, 1744.

Phenylboric Acid

$$C_6H_5B(OH)_2$$

$C_6H_7O_2B$ MW, 122

Needles from H_2O. M.p. 221° (216°). Sol. EtOH, Et_2O, hot H_2O. Volatile in steam.

Chlorine water ⟶ chlorobenzene. H_2O_2 ⟶ phenol. Boiling conc. HCl ⟶ benzene.

Ainley, Challenger, *J. Chem. Soc.*, 1930, 2171.
Seaman, Johnson, *J. Am. Chem. Soc.*, 1931, **53**, 713.
Bean, Johnson, *J. Am. Chem. Soc.*, 1932, **54**, 4417.
Krause, Nitsche, *Ber.*, 1922, **55**, 1264.
Krause, D.R.P., 371,467, (*Chem. Abstracts*, 1924, **18**, 992).

Phenylbromoacetic Acid.
See α-Bromophenylacetic Acid.

Phenyl ω-bromobenzyl Ketone.
See Desyl bromide.

4-Phenyl-1 : 2-butadiene (1-*Benzylallene*)

$C_6H_5{\cdot}CH_2{\cdot}CH{:}C{:}CH_2$

$C_{10}H_{10}$ MW, 130

B.p. 72–3°/17 mm. D_4^{20} 0·9220. n_D^{20} 1·5460.

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **55**, 2816.

1-Phenyl-1 : 3-butadiene (1-*Phenylerythrene*)

$CH_2{:}CH{\cdot}CH{:}CH{\cdot}C_6H_5$

$C_{10}H_{10}$ MW, 130

Cis :
B.p. 86°/11 mm. n^{28} 1·5950.
Trans :
B.p. 76°/11 mm. n^{28} 1·5920.

Muskat, Herrmann, *J. Am. Chem. Soc.*, 1931, **53**, 266.
Bergmann, Bondi, *Ber.*, 1931, **64**, 1479.

2-Phenyl-1 : 3-butadiene (2-*Phenylerythrene, phenoprene*)

$CH_2{:}CH{\cdot}C(C_6H_5){:}CH_2$

$C_{10}H_{10}$ MW, 130

B.p. 60–1°/17 mm. D_4^{20} 0·9226. n_D^{20} 1·5489.
Dimer : needles from MeOH. M.p. 62°. B.p. 220–5°/10 mm.

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **55**, 2816.

4 - Phenyl - 1 : 3 - butadiene - 1 - carboxylic Acid.
See 2-Styrylacrylic Acid.

4 - Phenyl - 1 : 3 - butadiene - 1 : 1 - dicarboxylic Acid.
See Cinnamylidenemalonic Acid.

Phenylbutandiol.
See Phenylbutylene Glycol *and* Phenyltetramethylene Glycol.

Phenylbutane.
See Butylbenzene.

Phenylbutenol.
See Methylstyrylcarbinol, Phenylallylcarbinol, Propenylphenylcarbinol *and* Allylphenylcarbinol.

1-Phenyl-*n*-butyl Alcohol.
See Propylphenylcarbinol.

2-Phenyl-*n*-butyl Alcohol (α-*Hydroxymethylpropylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2OH$

$C_{10}H_{14}O$ MW, 150

dl-.
B.p. 124–7°/25 mm., 120–1°/15 mm. D_4^{16} 0·989. n_D^{16} 1·43.
Benzoyl : m.p. 115–16°. B.p. 202–3°/18 mm.
Phenylurethane : m.p. 58–9°.
d-.
$[\alpha]_D^{25}$ + 1·8°, $[\alpha]_D^{30}$ + 2·54° (+ 1·11°).

Lagerev, *Chem. Zentr.*, 1935, II, 2809.
Levene, Marker, *J. Biol. Chem.*, 1935, **108**, 415.
Rampart, Amagat, *Ann. chim.*, 1927, **8**, 284.

3-Phenyl-*n*-butyl Alcohol

$CH_3{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_2OH$

$C_{10}H_{14}O$ MW, 150

l-.
B.p. 117°/8 mm. D_4^{25} 0·986. n_D^{25} 1·5201. $[\alpha]_D^{25}$ − 9·15° ($[\alpha]_D^{30}$ − 2·89°).
dl-.
B.p. 138–40°/33 mm., 125·5–128°/13 mm., 110–11°/6 mm. D_D^{20} 0·9834.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 762; 1935, **108**, 413.
Wojcik, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 4943.
Rupe, van Walraven, *Helv. Chim. Acta*, 1930, **13**, 369.

4-Phenyl-*n*-butyl Alcohol (δ-*Hydroxybutylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2OH$

$C_{10}H_{14}O$ MW, 150

Oil. B.p. 140°/14 mm. Insol. H_2O. D_4^{20} 1·001.
Me ether : $C_{11}H_{16}O$. MW, 164. B.p. 108°/11 mm.
Nitrite : $C_6H_5{\cdot}[CH_2]_4{\cdot}O{\cdot}NO$. B.p. 125–30°/15 mm.
p-*Nitrobenzoyl* : m.p. 18–20°.
Phenylurethane : m.p. 51–2°.

Kirner, *J. Am. Chem. Soc.*, 1926, **48**, 1112.
v. Braun, *Ber.*, 1911, **44**, 2871.
v. Braun, Deutsch, *Ber.*, 1912, **45**, 2176.
Normant, *Compt. rend.*, 1948, **226**, 733.

2-Phenyl-*sec*.-*n*-butyl Alcohol.
See Methylethylphenylcarbinol.

4-Phenyl-*sec*.-*n*-butyl Alcohol (γ-*Hydroxybutylbenzene, methyl*-β-*phenylethylcarbinol*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_3$

$C_{10}H_{14}O$ MW, 150

d-.
B.p. 132°/14 mm. D_4^{20} 0·9788. n_D^{20} 1·5168. Volatile in steam.
Formyl: b.p. 120°/15 mm. D_4^{20} 1·0083.
Acetyl: b.p. 130°/15 mm. D_4^{20} 0·9854.
Propionyl: b.p. 141°/16 mm. D_4^{20} 0·9790.

l-.
B.p. 132°/14 mm. n_D^{20} 1·5168. $[\alpha]_D^{20}$ − 19·45° in EtOH. Volatile in steam.

dl-.
Oil with aromatic odour. B.p. 238°/750 mm., 123–4°/15 mm. $D_4^{16\cdot5}$ 0·9899. $n_D^{16\cdot5}$ 1·517.
Acetyl: b.p. 123–4°/15 mm. D_4^{16} 0·991. n_D^{16} 1·4895.
Phenylurethane: m.p. 116–17° (113°).

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, **127**, 1094.
Zechmeister, Rom, *Ann.*, 1929, **468**, 126.
Schlenk, Bergmann, *Ann.*, 1930, **479**, 73.
Packendorff, *Ber.*, 1934, **67**, 906.
Pickard, Kenyon, *J. Chem. Soc.*, 1914, **105**, 1125.
Mozingo, Spencer, Folkers, *J. Am. Chem. Soc.*, 1944, **66**, 1860.

Phenyl-*tert*.-butyl Alcohol.
See Dimethylbenzylcarbinol.
1- *and* 4-Phenylbutylamines.
See Aminobutylbenzene.
2-Phenyl-*n*-butylamine

$$\begin{array}{c} C_6H_5 \\ | \\ CH_3{\cdot}CH_2{\cdot}CH{\cdot}CH_2NH_2 \end{array}$$

$C_{10}H_{15}N$ MW, 149

B.p. 110°/13 mm.
B,HCl: cryst. from Et_2O. M.p. 156°.

Rampart, Amagat, *Ann. chim.*, 1927, **8**, 285.

1-Phenyl-1-butylene (*α-Butenylbenzene*)

$$CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}C_6H_5$$

$C_{10}H_{12}$ MW, 132

B.p. 190–3° (186–7°), 89–90°/15 mm., 78°/12 mm., 70–1°/8 mm. D_4^{20} 0·9106. n_D^{16} 1·5381. Na + EtOH ⟶ butylbenzene.
Nitrosite: m.p. 124°.
Dibromide: m.p. 70°.

Auwers, Roth, Eisenlohr, *Ann.*, 1910, **373**, 282.
Klages, *Ber.*, 1904, **37**, 2312.
Lévy, Dvoleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1769.
For *cis*- and *trans*- forms see Muskat, Knapp, *Ber.*, 1931, **64**, 785.

4-Phenyl-1-butylene (*γ-Butenylbenzene*)

$$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}CH_2$$

$C_{10}H_{12}$ MW, 132

B.p. 177–8°/754 mm., 64°/10 mm. D_4^{20} 0·8831. n_D^{20} 1·5059. Heat of comb. C_v 1356·8 Cal. $KMnO_4$ in Me_2CO ⟶ hydrocinnamic acid.

André, *Bull. soc. chim.*, 1911, **9**, 193.
Rüber, *Ber.*, 1911, **44**, 2392.
Gilman, McGlumphy, *Bull. soc. chim.*, 1928, **43**, 1326.

1-Phenyl-2-butylene (*β-Butenylbenzene, crotonylbenzene*)

$$CH_3{\cdot}CH{:}CH{\cdot}CH_2{\cdot}C_6H_5$$

$C_{10}H_{12}$ MW, 132

Oily liq. B.p. 176°, 81–2°/21 mm., 70°/12 mm. D_4^{20} 0·8831. n_D^{20} 1·5101. Alc. KOH ⟶ 1-phenyl-1-butylene. Ozone in presence of H_2O ⟶ phenylacetaldehyde.

Auwers, Roth, Eisenlohr, *Ann.*, 1911, **385**, 108.
Klages, *Ber.*, 1904, **37**, 2310.
Straus, *Ann.*, 1905, **342**, 257.

2-Phenyl-2-butylene (*αβ-Dimethylstyrene, α-methylpropenylbenzene*)

$$\begin{array}{c} C_6H_5 \\ | \\ CH_3{\cdot}CH{:}C{\cdot}CH_3 \end{array}$$

$C_{10}H_{12}$ MW, 132

B.p. 187–9°, 80–1°/20 mm., 73°/14 mm. D_4^{25} 0·8911, D_4^{20} 0·9088. n_D^{20} 1·5339. Heat of comb. C_v 1352·8 Cal. $KMnO_4$ in H_2SO_4 ⟶ acetophenone.

Haller, Bauer, *Ann. chim.*, 1918, **9**, 12.
Klages, *Ber.*, 1902, **35**, 2641, 3508.
Broun, Voronkov, *J. Gen. Chem. U.S.S.R.* 1947, **17**, 1162, (*Chem. Abstracts*, 1948, **42**, 1591).

1-Phenyl-α-butylene Glycol (*1-Phenyl-*1 : 2-*butandiol, αβ-dihydroxybutylbenzene*)

$$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CH(OH){\cdot}C_6H_5$$

$C_{10}H_{14}O_2$ MW, 166

Two forms. (i) M.p. 40–1°. (ii) B.p. 205–8°/72 mm., 143°/9 mm. D_4^{17} 1·04. n_D^{17} 1·538.

Lévy, Dvoleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1774.

2-Phenyl-α-butylene Glycol (*2-Phenyl-*1 : 2-*butandiol*)

$$\begin{array}{c} C_6H_5 \\ | \\ CH_3{\cdot}CH_2{\cdot}C(OH){\cdot}CH_2OH \end{array}$$

$C_{10}H_{14}O_2$ MW, 166

Cryst. from ligroin. M.p. 56°. B.p. 165°/23 mm. Sol. H_2O, EtOH, Et_2O. At 250–300° ⟶ diphenylacetaldehyde. At 400–50° ⟶ deoxybenzoin.

Ramart-Lucas, Salmon-Legagneur, *Bull. soc. chim.*, 1932, **51**, 1078.
Stoermer *et al.*, *Ber.*, 1906, **39**, 2300.

4-Phenyl-α-butylene Glycol (*4-Phenyl-1 : 2-butandiol, γδ-dihydroxybutylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2OH$

$C_{10}H_{14}O_2$ MW, 166

Viscous oil. B.p. 180°/13 mm.

v. Braun, *Ber.*, 1923, **56**, 2182.

1-Phenyl-β-butylene Glycol (*1-Phenyl-1 : 3-butandiol, αγ-dihydroxybutylbenzene*)

$CH_3{\cdot}CH(OH){\cdot}CH_2{\cdot}CH(OH){\cdot}C_6H_5$

$C_{10}H_{14}O_2$ MW, 166

Powder. Sinters at 60°. M.p. about 73·5°. B.p. 175–8°/21 mm., 168–9°/13 mm., 129–31°/2 mm. Sol. EtOH, C_6H_6, ligroin, hot Et_2O. Spar. sol. H_2O.

Diacetyl: b.p. 157°/10 mm., 140°/2 mm. Decomp. on dist. at ord. press.

Michael, Ross, *J. Am. Chem. Soc.*, 1931, **53**, 2412.
Sprague, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 2670.
Bauer, *Compt. rend.*, 1912, **154**, 1093.

3-Phenyl-β-butylene Glycol (*3-Phenyl-1 : 3-butandiol*)

$CH_3{\cdot}C(C_6H_5)(OH){\cdot}CH_2{\cdot}CH_2OH$

$C_{10}H_{14}O_2$ MW, 166

B.p. 130°/1 mm. D^{20} 1·0865. n_D^{20} 1·5341.

Diacetyl: b.p. 142°/0·5 mm.

Di-p-nitrobenzoyl: m.p. 104°.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1932, **15**, 1266.

1-Phenyl-1-butyne (*1-Ethyl-2-phenylacetylene*)

$CH_3{\cdot}CH_2{\cdot}C{\vdots}C{\cdot}C_6H_5$

$C_{10}H_{10}$ MW, 130

B.p. 201–3°, 87–90°/18 mm. D^{18} 0·915. n_D^{18} 1·537.

Truchet, *Ann. chim.*, 1931, **16**, 397.
Morgan, *J. Chem. Soc.*, 1876, **29**, 162.
Bergmann, Bondi, *Ber.*, 1933, **66**, 286.

4-Phenyl-1-butyne (*β-Phenylethylacetylene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}C{\vdots}CH$

$C_{10}H_{10}$ MW, 130

B.p. 189–91°/758 mm., 83°/15 mm. D_4^0 0·928, D_4^{14} 0·918. n_D^{20} 1·5212. Heat of comb. C_v 1340·1 Cal.

André, *Bull. soc. chim.*, 1911, **9**, 193.
Moureu, André, *Ann. chim.*, 1914, **1**, 116.
Bert, Dorier, Lamy, *Compt. rend.*, 1925, **181**, 555.

1-Phenylbutyraldehyde (*Ethylphenylacetaldehyde*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CHO$

$C_{10}H_{12}O$ MW, 148

Oil. B.p. 211°, 104–6°/15 mm.

Semicarbazone: m.p. 155°.

Stoermer, *Ber.*, 1906, **39**, 2300.

2-Phenylbutyraldehyde.

See β-Methylhydrocinnamaldehyde.

3-Phenylbutyraldehyde

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CHO$

$C_{10}H_{12}O$ MW, 148

B.p. 129–31°/17 mm. (120–2°/16 mm.).

Semicarbazone: leaflets from MeOH. M.p. 104–5°.

Di-Me acetal: b.p. 121–4°/9 mm.

v. Braun, Anton, Fischer, Keller, Manz, *Ber.*, 1934, **67**, 225.
v. Braun, Kruber, *Ber.*, 1912, **45**, 394.

1-Phenylbutyric Acid (*Ethylphenylacetic acid*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}COOH$

$C_{10}H_{12}O_2$ MW, 164

Plates from Et_2O. M.p. 42°. B.p. 270°.

Me ester: $C_{11}H_{14}O_2$. MW, 178. B.p. 228°.

Amide: $C_{10}H_{13}ON$. MW, 163. M.p. 86°.

Nitrile: $C_{10}H_{11}N$. MW, 145. B.p. 238–40°, 114–15°/15 mm. D^{14} 0·977. Sol. EtOH, C_6H_6. Insol. H_2O. Volatile in steam.

Riiber, *Ber.*, 1903, **36**, 1406.
Rupe, *Ann.*, 1909, **369**, 334.
Neure, *Ann.*, 1889, **250**, 154.
Bodroux, Taboury, *Bull. soc. chim.*, 1910, **7**, 667.

2-Phenylbutyric Acid.

See β-Methylhydrocinnamic Acid.

3-Phenylbutyric Acid (*2-Benzylpropionic acid*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_{10}H_{12}O_2$ MW, 164

Leaflets from H_2O. M.p. 52°. B.p. 290°, 171°/15 mm. Sol. EtOH, Et_2O. Mod. sol. warm H_2O.

Et ester: $C_{12}H_{16}O_2$. MW, 192. B.p. 130–1°/10 mm. D_4^{20} 1·001.

l-Menthyl ester: b.p. 205°/10 mm. $[\alpha]_D^{20}$ − 57·0° in C_6H_6.

Chloride: $C_{10}H_{11}OCl$. MW, 182·5. B.p. 140–2°/12 mm., 119°/9 mm.

Amide: $C_{10}H_{13}ON$. MW, 163. Plates from H_2O. M.p. 84·5°. Sol. EtOH, Et_2O.

Nitrile: $C_{10}H_{11}N$. MW, 145. B.p. 142–5°/16 mm., 129–31°/10 mm.

Overbaugh, Allen, Martin, Fieser, *Organic Syntheses*, 1935, XV, 64.
Richard, *Compt. rend.*, 1935, **200**, 1945.
Amagat, *Bull. soc. chim.*, 1927, **41**, 942.
Auwers, Möller, *J. prakt. Chem.*, 1925, **109**, 138.
v. Braun, *Ber.*, 1910, **43**, 2844; 1911, **44**, 2871.
Rupe, *Ann.*, 1913, **395**, 118.

Phenylbutyrolactam.
See Phenylpyrrolidone.

3-Phenylbutyrolactone.
See under 3-Hydroxy-3-phenylbutyric Acid.

3-Phenyl-γ-butyrolactone-2-carboxylic Acid.
See γ-Phenylparaconic Acid.

Phenylcacodyl (*Tetraphenyldiarsine*)

$(C_6H_5)_2As{\cdot}As(C_6H_5)_2$

$C_{24}H_{20}As_2$ MW, 458

Cryst. M.p. 135°. B.p. 200°/1 mm. Sol. EtOH. Less sol. Et_2O. Sol. 100 parts C_6H_6 at 20°.

Michaelis, *Ann.*, 1902, **321**, 148.
Michaelis, Schulte, *Ber.*, 1882, **15**, 1954.
Porter, Borgstrom, *J. Am. Chem. Soc.*, 1919, **41**, 2049.

5-Phenylcapric Acid

$CH_3{\cdot}[CH_2]_3{\cdot}CH(C_6H_5){\cdot}[CH_2]_4{\cdot}COOH$

$C_{16}H_{24}O_2$ MW, 248

B.p. 176–80°/3 mm. D_{25}^{25} 0·9817. n_D^{20} 1·5000.
p-*Bromophenacyl ester*: m.p. 55·5–56°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2523.

9-Phenylcapric Acid

$C_6H_5{\cdot}CH_2{\cdot}[CH_2]_7{\cdot}CH_2{\cdot}COOH$

$C_{16}H_{24}O_2$ MW, 248

Waxy solid. M.p. 41°. B.p. 228–30°/18 mm. Very sol. usual org. solvents.
Et ester: $C_{18}H_{28}O_2$. MW, 276. B.p. 220–4°/20 mm.

Borsche, *Ber.*, 1919, **52**, 2085.

1-Phenylcaproic Acid

$CH_3{\cdot}[CH_2]_3{\cdot}CH(C_6H_5){\cdot}COOH$

$C_{12}H_{16}O_2$ MW, 192

B.p. 182–3°/20 mm. D_4^{19} 1·0225. n_D^{19} 1·5071.
Amide: $C_{12}H_{17}ON$. MW, 191. M.p. 96°.
Nitrile: $C_{12}H_{15}N$. MW, 173. B.p. 151·1–152·5°/20 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.
Dolique, *Ann. chim.*, 1931, **15**, 468.

2-Phenylcaproic Acid

$CH_3{\cdot}[CH_2]_2{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}COOH$

$C_{12}H_{16}O_2$ MW, 192

d-.
B.p. 152°/4 mm. D_4^{25} 1·025. n_D 1·5078. $[\alpha]_D^{25}$ + 5·93°.
Et ester: $C_{14}H_{20}O_2$. MW, 220. B.p. 123°/2 mm. D_4^{25} 0·969. n_D^{25} 1·4870. $[\alpha]_D^{25}$ + 3·45°.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 765.

3-Phenylcaproic Acid

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_{12}H_{16}O_2$ MW, 192

dl-.
Cryst. M.p. 104·5–105·5°. B.p. 185°/22 mm.
Chloride: $C_{12}H_{15}OCl$. MW, 210·5. B.p. 138°/18 mm.
Nitrile: $C_{12}H_{15}N$. MW, 173. B.p. 148–50°/19 mm.

l-.
B.p. 156°/4 mm. $[\alpha]_D^{25}$ − 0·61°.

Lévy, *Compt. rend.*, 1933, **197**, 772.
Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 329.

4-Phenylcaproic Acid

$CH_3{\cdot}CH(C_6H_5){\cdot}[CH_2]_2{\cdot}CH_2{\cdot}COOH$

$C_{12}H_{16}O_2$ MW, 192

d-.
B.p. 156°/1 mm. D_4^{25} 1·022. $[\alpha]_D^{25}$ + 2·01°.
Et ester: $C_{14}H_{20}O_2$. MW, 220. B.p. 145°/1 mm. D_4^{25} 0·974. $[\alpha]_D^{25}$ + 1·40°.

dl-.
B.p. 173–5°/11 mm.
Chloride: $C_{12}H_{15}OCl$. MW, 210·5. B.p. 134–6°/13 mm.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 771.
Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 271.

5-Phenylcaproic Acid

$C_6H_5{\cdot}CH_2{\cdot}[CH_2]_3{\cdot}CH_2{\cdot}COOH$

$C_{12}H_{16}O_2$ MW, 192

Cryst. M.p. 22–4° (11°). B.p. 206–8°/30 mm., 180–90°/17 mm. (186–8°/11 mm.).
Et ester: $C_{14}H_{20}O_2$. MW, 220. B.p. 162–4°/12 mm.
Chloride: $C_{12}H_{15}OCl$. MW, 210·5. B.p. 151–2°/11 mm.
Nitrile: $C_{12}H_{15}N$. MW, 173. B.p. 161–3°/13 mm.
Anilide: m.p. 80°.
p-*Toluidide*: m.p. 78°.
Piperidide: b.p. 177°/0·03 mm.

v. Braun, *Ber.*, 1911, **44**, 2873.
Borsche, *Ber.*, 1919, **52**, 2084.
Staudinger, Müller, *Ber.*, 1923, **56**, 714.
Grateau, *Compt. rend.*, 1930, **191**, 947.

4-Phenylcaprylic Acid

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}[CH_2]_3{\cdot}COOH$

$C_{14}H_{20}O_2$ MW, 220

l-.
B.p. 170°/1 mm. D_4^{25} 0·999. $[\alpha]_D^{25}$ − 0·82°.

Et ester: $C_{16}H_{24}O_2$. MW, 248. B.p. 152°/1 mm. D_4^{25} 0·958. $[\alpha]_D^{25}$ − 0·49°.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 770.

7-Phenylcaprylic Acid

$C_6H_5 \cdot CH_2 \cdot [CH_2]_5 \cdot CH_2 \cdot COOH$

$C_{14}H_{20}O_2$ MW, 220

Leaflets. M.p. about 30°. B.p. 209–10°/14 mm.

Borsche, *Ber.*, 1919, **52**, 2084.

Phenylcarbamic Acid (*Carbanilic acid*)

$C_6H_5 \cdot NH \cdot COOH$

$C_7H_7O_2N$ MW, 137

Not known in free state.

Me ester: phenylurethylan. $C_8H_9O_2N$. MW, 151. Leaflets from EtOH. M.p. 47°.

Et ester: *see* Phenylurethane.

Propyl ester: $C_{10}H_{13}O_2N$. MW, 179. Needles. M.p. 57–9°.

Isopropyl ester: needles from EtOH. M.p. 75–6° (90°).

Butyl ester: $C_{11}H_{15}O_2N$. MW, 193. Leaflets or prisms from pet. ether. M.p. 65·5° (57°).

Isobutyl ester: needles. M.p. 86°.

tert.-*Butyl ester*: needles from pet. ether. M.p. 136° (108–108·5°).

dl-*Amyl ester*: $C_{12}H_{17}O_2N$. MW, 207. M.p. 31°.

d-*Amyl ester*: cryst. from ligroin. M.p. 30°.

Isoamyl ester: cryst. from pet. ether. M.p. 55°.

tert.-*Amyl ester*: m.p. 42°.

Allyl ester: cryst. M.p. 70°.

l-*Menthyl ester*: needles from EtOH. M.p. 111–12°. $[\alpha]_D^{20}$ − 77·21° in $CHCl_3$.

dl-*Menthyl ester*: needles from MeOH.Aq. M.p. 104°.

Tropine ester: *see* Uretropine.

p-*Chlorophenyl ester*: m.p. 138–9°.

Benzyl ester: m.p. 77°.

Amide: *see* Phenylurea.

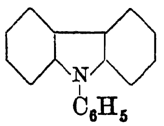
Anilide: *see* Carbanilide.

Nitrile: *see* Phenylcyanamide.

Jeffreys, *Am. Chem. J.*, 1901, **22**, 18.
Roemer, *Ber.*, 1873, **6**, 1103.
Lambling, *Bull. soc. chim.*, 1898, **19**, 777.
Piccard, Littlebury, *J. Chem. Soc.*, 1912, **101**, 116.
Weizmann, Garrard, *J. Chem. Soc.*, 1920, **117**, 328.

Phenylcarbazinic Acid.
See Phenylhydrazine-β-carboxylic Acid.

***N*-Phenylcarbazole**



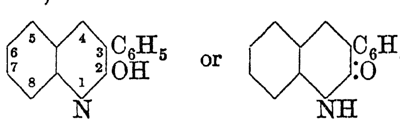
$C_{18}H_{13}N$ MW, 243

Needles or plates from EtOH. M.p. 94–5° (82–4°).

Picrate: red needles from ligroin. M.p. 126–9°.

Cassella, D.R.P., 224,951, (*Chem. Zentr.*, 1910, II, 699).
Eckert, Seidel, Endler, *J. prakt. Chem.*, 1922, **104**, 88.
Montmollin, Montmollin, *Helv. Chim. Acta*, 1923, **6**, 98.
Hager, *Organic Syntheses*, Collective Vol. I, 532 (*Note* 13).
Barclay, Campbell, *J. Chem. Soc.*, 1945, 530.

3-Phenylcarbostyril (2-*Hydroxy*-3-*phenylquinoline*)



$C_{15}H_{11}ON$ MW, 221

Needles from EtOH. M.p. 234–5°. Sol. hot EtOH, C_6H_6. Insol. Et_2O.

Bischler, Lang, *Ber.*, 1895, **28**, 292.
Stoermer, Prigge, *Ann.*, 1915, **409**, 27.
Wislicenus, Erbe, *Ann.*, 1920, **421**, 146.

4-Phenylcarbostyril (2-*Hydroxy*-4-*phenylquinoline*).

Needles. M.p. 259°. Sol. AcOH, hot C_6H_6. Spar. sol. hot H_2O, EtOH, $CHCl_3$, CS_2, ligroin. Zn + H ⟶ 4-phenylquinoline.

Camps, *Arch. Pharm.*, 1899, **237**, 683.

Phenyl *o*-carboxybenzyl Ketone.
See *o*-Phenacylbenzoic Acid.

Phenyl-carboxymethyl-glycine.
See 1-Phenyliminodiacetic Acid.

Phenyl-ω-carboxysarcosine.
See 1-Phenyliminodiacetic Acid.

Phenylcarbylamine.
See Phenyl isocyanide.

Phenylcarbylamine chloride.
See Phenyliminophosgene.

Phenylchloroacetic Acid (1-*Chloro*-1-*phenylacetic acid*)

$C_6H_5 \cdot CHCl \cdot COOH$

$C_8H_7O_2Cl$ MW, 170·5

d-.

Cryst. from pet. ether. M.p. 60–1°. $[\alpha]_D^{20}$ + 191·9° in C_6H_6.

Me ester: $C_9H_9O_2Cl$. MW, 184·5. B.p. 178°/40 mm., 124°/8 mm. D_4^{20} 1·1882. $[\alpha]_D^{18}$ + 25·67°.

Et ester: $C_{10}H_{11}O_2Cl$. MW, 198·5. B.p. 162°/45 mm., 138°/19 mm., 133°/12 mm. D_4^{20} 1·1594. n_D^{20} 1·5152. $[\alpha]_D^{20}$ + 121·0° in EtOH.

Propyl ester: $C_{11}H_{13}O_2Cl$. MW, 212·5.

B.p. 180°/60 mm., 140°/19 mm. D_4^{20} 1·1278. n_D^{20} 1·5095. $[\alpha]_D^{18}$ + 23·94°.

Butyl ester: $C_{12}H_{15}O_2Cl$. MW, 226·5. B.p. 163–4°/20 mm. D^{20} 1·1040.

l-*Menthyl ester*: needles from EtOH.Aq. M.p. 56–7°. $[\alpha]_D^{15\cdot5}$ + 5·7° in EtOH.

Chloride: $C_8H_6OCl_2$. MW, 189. B.p. 120°/23 mm. $[\alpha]_D^{18}$ + 158·33° in CS_2.

l-.

Needles from pet. ether. M.p. 61°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. cold H_2O. $[\alpha]_D^{18}$ − 163° in EtOH, − 191·3° in C_6H_6.

Me ester: b.p. 123–6°/11 mm. D_4^{18} 1·213. $[\alpha]_D^{15}$ − 86·7°.

Et ester: b.p. 132–3°/15 mm. $D_4^{16\cdot4}$ 1·162. $[\alpha]_D^{20}$ − 108° in EtOH.

l-*Menthyl ester*: needles from EtOH.Aq. M.p. 44·5–45·5°. $[\alpha]_D^{15}$ − 150·1°.

Chloride: b.p. 103°/11 mm. $[\alpha]_{578}^{20}$ − 250°.

Dimethylamide: m.p. 47°. $[\alpha]_{578}$ − 110° in EtOH.

dl-.

Leaflets. M.p. 78°. Very sol. EtOH, Et_2O. Mod. sol. hot ligroin. Spar. sol. H_2O.

Me ester: b.p. 248° decomp.

Et ester: b.p. 142°/19 mm.

l-*Menthyl ester*: cryst. from Et_2O. M.p. 28–9°. B.p. 200–1°/12 mm. D_4^{20} 1·064. $[\alpha]_D^{20}$ − 67·2°.

Chloride: b.p. 120°/15 mm., 110°/4 mm.

Amide: C_8H_8ONCl. MW, 169·5. Needles from C_6H_6. M.p. 116°. Sol. EtOH, Et_2O.

Nitrile: C_8H_6NCl. MW, 151·5. B.p. 131·5°/13 mm., 108–10°/5 mm.

McKenzie, Clough, *J. Chem. Soc.*, 1909, **95**, 784.
Walden, *Ber.*, 1895, **28**, 1295.
Bischoff, Walden, *Ann.*, 1894, **279**, 122.
Meyer, Boner, *Ann.*, 1883, **220**, 44.
Michaël, Jeanprêtre, *Ber.*, 1892, **25**, 1680.
Darapsky, *J. prakt. Chem.*, 1919, **99**, 188.
McKenzie, Barrow, *J. Chem. Soc.*, 1911, **99**, 1916.
McKenzie, Smith, *J. Chem. Soc.*, 1923, **123**, 1962.
Freudenberg, Todd, Seidler, *Ann.*, 1933, **501**, 217.
Barrow, Thorneycroft, *J. Chem. Soc.*, 1934, 724.
Hignett, Kay, *J. Soc. Chem. Ind.*, 1935, **54**, 98T.

Phenyl-chlorobenzylamine.
See under Chlorobenzylamine.

Phenyl chlorobenzyl Ketone.
See Chlorodeoxybenzoin.

Phenyl ω-chlorobenzyl Ketone.
See Desyl chloride.

Phenylchloroform.
See Benzotrichloride.

Phenyl-2-chlorolactic Acid (2-*Chloro*-1-*hydroxy*-2-*phenylpropionic acid, cinnamic acid chlorohydrin*)

$C_6H_5 \cdot CHCl \cdot CH(OH) \cdot COOH$

$C_9H_9O_3Cl$ MW, 200·5

l-.

Cryst. M.p. 144°. $[\alpha]_D$ − 71·7°.

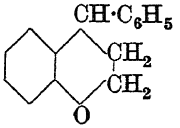
dl-.

Needles from $CHCl_3$. M.p. 141–2°. Sol. EtOH, Et_2O. Spar. sol. $CHCl_3$, C_6H_6, ligroin. Hot $H_2O \longrightarrow CO_2 + HCl +$ phenylacetaldehyde. NaOH ⟶ phenylglycidic acid.

Erlenmeyer, *Ann.*, 1892, **271**, 150; *Ber.*, 1906, **39**, 789.

2-Phenylchroman.
See Flavan.

4-Phenylchroman



$C_{15}H_{14}O$ MW, 210

Needles from pet. ether. M.p. 38·5°. Addn. of $FeCl_3$ to suspension in H_2SO_4 ⟶ reddish-violet col.

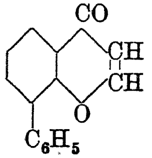
Greenwood, Nierenstein, *J. Chem. Soc.*, 1920, **117**, 1597.

2-Phenylchromanone.
See Flavanone.

2-Phenylchromone.
See Flavone.

3-Phenylchromone.
See Isoflavone.

8-Phenylchromone

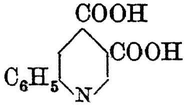


$C_{15}H_{10}O_2$ MW, 222

Needles from EtOH.Aq. M.p. 112°.

Watson, *J. Chem. Soc.*, 1916, **109**, 305.

6-Phenylcinchomeronic Acid (6-*Phenylpyridine*-3 : 4-*dicarboxylic acid*)



$C_{13}H_9O_4N$ MW, 243

Yellow needles and prisms from H_2O. M.p. 248–50°. Spar. sol. H_2O, Et_2O. Mod. sol. EtOH.

Di-Me ester: $C_{15}H_{13}O_4N$. MW, 271. Plates from $CHCl_3$. M.p. 74°.

Boehm, Bournot, *Ber.*, 1915, **48**, 1573.

Phenylcinchoninic Acid.
See Phenylquinoline-4-carboxylic Acid.

α-Phenylcinnamaldehyde (1 : 2-*Diphenylacrolein, α-formylstilbene*)

C_6H_5
$C_6H_5·CH{:}C·CHO$

$C_{15}H_{12}O$ MW, 208

Cryst. from EtOH. M.p. 94°. B.p. 195–200°/17 mm. Ag_2O in C_6H_6 ⟶ 1-phenylcinnamic acid.
Oxime: leaflets from EtOH. M.p. 165–6°.
Semicarbazone: cryst. from EtOH. M.p. 188·5–189·5°.
Phenylhydrazone: yellow needles from AcOH. M.p. 125–6°.
2 : 4-*Dinitrophenylhydrazone*: m.p. 240–1°.

Meerwein, *J. prakt. Chem.*, 1918, **97**, 281.
Shoruigan, Isagulyantz, Machinskaya, *Ber.*, 1933, **66**, 389.

β-Phenylcinnamaldehyde (2 : 2-*Diphenylacrolein*)

$(C_6H_5)_2C{:}CH·CHO$

$C_{15}H_{12}O$ MW, 208

Pale yellow prisms from ligroin. M.p. 44°.
Semicarbazone: m.p. 214–15°.
Phenylhydrazone: yellow needles. M.p. 173°.

Ziegler, Tiemann, *Ber.*, 1922, **55**, 3413.
Kohler, Larsen, *J. Am. Chem. Soc.*, 1935, **57**, 1452.

α-Phenylcinnamic Acid (*Stilbene-α-carboxylic acid,* 1 : 2-*diphenylacrylic acid*)

C_6H_5
$C_6H_5·CH{:}C·COOH$

$C_{15}H_{12}O_2$ MW, 224

Trans:
Needles from ligroin or EtOH.Aq. M.p. 172°. Sol. EtOH, Et_2O. Mod. sol. hot H_2O. Irradiation ⟶ *cis*-form.
Me ester: $C_{16}H_{14}O_2$. MW, 238. Needles from EtOH.Aq. M.p. 77–8°.
Et ester: $C_{17}H_{16}O_2$. MW, 252. Cryst. M.p. 33–4° (28°). B.p. 214–15°/28 mm., 192°/13 mm. $D_4^{18·2}$ 1·0971. $n_D^{18·6}$ 1·5972.
l-*Menthyl ester*: needles or leaflets from EtOH.Aq. $[\alpha]_D^{20}$ − 53·44° in C_6H_6.
Phenyl ester: $C_{21}H_{16}O_2$. MW, 300. Needles from EtOH–$CHCl_3$. M.p. 142°. Sol. $CHCl_3$. Mod. sol. Me_2CO, C_6H_6. Spar. sol. EtOH, Et_2O.
o-*Tolyl ester*: $C_{22}H_{18}O_2$. MW, 314. Needles from EtOH. M.p. 130°.
Amide: $C_{15}H_{13}ON$. MW, 223. Needles from Me_2CO.Aq. M.p. 127°.
Nitrile: $C_{15}H_{11}N$. MW, 205. Cryst. from EtOH. M.p. 49–51°. B.p. 213–14°/23 mm.
Anilide: cryst. from EtOH. M.p. 141°.
Cis:
Leaflets from EtOH. M.p. 137–8°. More sol. than *trans*- form.
Amide: cryst. from $CHCl_3$–ligroin. M.p. 167–8°.
Nitrile: cryst. from EtOH. M.p. 86°. B.p. 228–30°/23 mm.
Anilide: cryst. from $CHCl_3$–pet. ether. M.p. 179°.

Stoermer, Voht, *Ann.*, 1915, **409**, 39.
Cabella, *Gazz. chim. ital.*, 1884, **14**, 114.
Rupe, *Ann.*, 1909, **369**, 315.
Walther, *J. prakt. Chem.*, 1896, **53**, 454.
v. Braun, Manz, *Ann.*, 1929, **468**, 258.
Pfeiffer, Engelhardt, Alfuss, *Ann.*, 1928, **467**, 158.
Müller, Gawlich, Krentzmann, *Ann.*, 1935, **515**, 111.
Buckles, Hausman, *J. Am. Chem. Soc.*, 1948, **70**, 415.

β-Phenylcinnamic Acid (2 : 2-*Diphenylacrylic acid*)

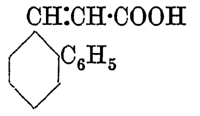
$(C_6H_5)_2C{:}CH·COOH$

$C_{15}H_{12}O_2$ MW, 224

Leaflets from EtOH. M.p. 162° (167°). Sol. EtOH, Et_2O, AcOH. Spar. sol. H_2O, ligroin.
Me ester: oil. B.p. 194·6–194·8°/13 mm.
Et ester: b.p. 207°/17 mm.
l-*Menthyl ester*: needles from EtOH. M.p. 66–7°. $[\alpha]_D^{20}$ − 37·92° in C_6H_6.
Nitrile: pale yellow needles from MeOH. M.p. 49°.
Anilide: m.p. 130–1°.
Anhydride: $C_{30}H_{22}O_3$. MW, 430. Needles from EtOH. M.p. 118–20°. Cold conc. H_2SO_4 ⟶ green col. ⟶ red on heating.

de Fazi, *Gazz. chim. ital.*, 1915, **45**, ii, 5.
Rupe, *Ann.*, 1909, **369**, 315; 1913, **395**, 142.
Kohler, Reimer, *Am. Chem. J.*, 1905, **33**, 343.
Rupe, Busolt, *Ber.*, 1907, **40**, 4539.
Posner, *J. prakt. Chem.*, 1910, **82**, 439.
Schlenk, Bergmann, *Ann.*, 1928, **463**, 237.
Bergmann *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 1612.

o-Phenylcinnamic Acid



$C_{15}H_{12}O_2$ MW, 224

Cryst. from EtOH. M.p. 202°. Sol. Et_2O, EtOH, $CHCl_3$.

Zaheer, Faseeh, *J. Indian Chem. Soc.*, 1944, **21**, 381.

p-Phenylcinnamic Acid.
Needles from 60% AcOH. M.p. 225°.
Et ester: $C_{17}H_{16}O_2$. MW, 252. M.p. 87°.

v. Braun, Nelles, *Ber.*, 1933, **66**, 1465.
Vorländer, *Ber.*, 1935, **68**, 453.
Hey, *J. Chem. Soc.*, 1931, 2478.

Phenylcitraconic Acid (*Benzylmaleic acid*)

$C_6H_5{\cdot}CH_2{\cdot}C{\cdot}COOH$
$HC{\cdot}COOH$

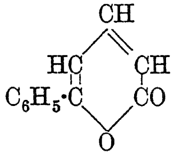
$C_{11}H_{10}O_4$ MW, 206

Cryst. from Et_2O–ligroin. M.p. 105–8°. Very sol. H_2O, Et_2O. Mod. sol. $CHCl_3$. Insol. CS_2, ligroin. Hot $H_2O \longrightarrow$ phenylitaconic acid. 10% NaOH $\longrightarrow$ phenylitaconic + phenylaticonic acids. Br in sunlight $\longrightarrow$ phenylmesaconic acid.

Anhydride: $C_{11}H_8O_3$. MW, 188. Prisms from Et_2O. M.p. 60–1°. Sol. C_6H_6, warm Et_2O, boiling CS_2. Spar. sol. boiling ligroin. $H_2O \longrightarrow$ phenylcitraconic acid.

Fittig, Brooke, *Ann.*, 1899, **305**, 21.

6-Phenylcoumalin (*6-Phenyl-α-pyrone*)



$C_{11}H_8O_2$ MW, 172

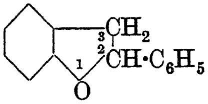
Present in Coto bark. Needles from pet. ether or MeOH.Aq. M.p. 68°. Sol. EtOH. Et_2O, $CHCl_3$, AcOH. Mod. sol. warm pet. ether.

Picrate: yellow plates from Et_2O. M.p. 81–2°.

Dimer: cryst. powder. M.p. 219° decomp. Spar. sol. boiling EtOH, Me_2CO. Insol. Et_2O, AcOH, C_6H_6.

Severni, *Gazz. chim. ital.*, 1896, **26**, ii, 338.
Ciamician, Silber, *Ber.*, 1894, **27**, 841.
Kalf, *Rec. trav. chim.*, 1927, **46**, 594.

2-Phenylcoumaran



$C_{14}H_{12}O$ MW, 196

Cryst. M.p. 32–3°. Volatile in steam. Conc. $H_2SO_4 \longrightarrow$ pale yellow col.

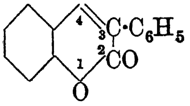
Stoermer, Reuter, *Ber.*, 1903, **36**, 3983.

3-Phenylcoumaran.

Needles from EtOH. M.p. 38·5°. B.p. 167°/14 mm. Sol. in conc. H_2SO_4 turns yellow on standing. Conc. $H_2SO_4 + FeCl_3 \longrightarrow$ brownish-green col.

Stoermer, Reuter, *Ber.*, 1903, **36**, 3984.
Stoermer, Kippe, *Ber.*, 1903, **36**, 4006, 4008.

3-Phenylcoumarin



$C_{15}H_{10}O_2$ MW, 222

Needles from AcOH. M.p. 140–1°.

Semicarbazone: pale yellow cryst. Decomp. at 210–13°.

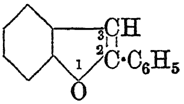
Lovett, Roberts, *J. Chem. Soc.*, 1928, 1977.
Stoermer, Prigge, *Ann.*, 1915, **409**, 27.

4-Phenylcoumarin.

Needles from EtOH or H_2O. M.p. 105°. Sol. most org. solvents. Spar. sol. boiling H_2O. Sol. conc. H_2SO_4.

Stoermer, Friderici, *Ber.*, 1908, **41**, 340.

2-Phenylcoumarone



$C_{14}H_{10}O$ MW, 194

Leaflets from EtOH.Aq. M.p. 120–1°. Volatile in steam. Sol. conc. H_2SO_4 with yellow col. showing blue fluor. on heating.

Stoermer, Reuter, *Ber.*, 1903, **36**, 3981.
v. Kostanecki, Tambor, *Ber.*, 1909, **42**, 826.
Shin'iti Kuwai *et al.*, *Chem. Abstracts*, 1939, **33**, 5394.

3-Phenylcoumarone.

Cryst. M.p. 42°. B.p. 316–17°, 177–8°/15 mm. D^{19} 1·1449. Sol. conc. H_2SO_4 with orange-yellow col. showing blue fluor. on heating.

Stoermer, Kippe, *Ber.*, 1903, **36**, 4004.

1-Phenylcrotonaldehyde

C_6H_5
$CH_3{\cdot}CH{:}C{\cdot}CHO$

$C_{10}H_{10}O$ MW, 146

B.p. 117°/15 mm. D_0^{18} 1·045. n_D^{18} 1·5605.

Oxime: m.p. 116°.

Semicarbazone: m.p. 201°.

Tiffeneau, Weill, *Compt. rend.*, 1935, **200**, 1217.

2-Phenylcrotonaldehyde.

See β-Methylcinnamaldehyde.

3-Phenylcrotonaldehyde

$C_6H_5{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CHO$

$C_{10}H_{10}O$ MW, 146

Semicarbazone: m.p. 210°.

Prévost, *Bull. soc. chim.*, 1944, **11**, 218.

1-Phenylcrotonic Acid (*α-Ethylidenephenylacetic acid*, *β-methylatropic acid*)

C_6H_5
$CH_3{\cdot}CH{:}C{\cdot}COOH$

$C_{10}H_{10}O_2$ MW, 162

Prisms from EtOH, needles or plates from H_2O. M.p. 136°. Spar. sol. cold H_2O. $KMnO_4 \longrightarrow$ benzoylformic acid + acetaldehyde.

Et ester: $C_{12}H_{14}O_2$. MW, 190. B.p. 128–31°/15 mm. Volatile in steam.

l-*Menthyl ester*: yellow oil. Decomp. on dist. $[\alpha]_D^{20}$ − 46·13° in C_6H_6.

Amide: $C_{10}H_{11}ON$. MW, 161. Plates from C_6H_6. M.p. 98–9°.

Nitrile: $C_{10}H_9N$. MW, 143. B.p. 224–6°/751 mm., 125°/13–14 mm. D_4^{20} 1·013. n_D^{20} 1·5555.

Rupe, Busolt, *Ann.*, 1909, **369**, 332.
Dimroth, Feuchter, *Ber.*, 1903, **36**, 2253.
Knowles, Cloke, *J. Am. Chem. Soc.*, 1932, **54**, 2028.
Pfeiffer, Engelhardt, Alfuss, *Ann.*, 1928, **467**, 158.
Ray, *Chem. Abstracts*, 1928, **22**, 3647.

2-Phenylcrotonic Acid.
See β-Methylcinnamic Acid.

3-Phenylcrotonic Acid (2-*Benzylacrylic acid*)

$C_6H_5·CH_2·CH{:}CH·COOH$

$C_{10}H_{10}O_2$ MW, 162

Two forms. (i) Plates from C_6H_6, pearly leaflets from H_2O. M.p. 65°. Sol. EtOH, Et_2O, C_6H_6, CS_2. (ii) Liq. Passes into (i) on heating with dil. HCl.

Bougault, *Compt. rend.*, 1917, **164**, 635.
Fittig, Luib, *Ann.*, 1894, **283**, 55, 302.
Vorländer, Strunck, *Ann.*, 1906, **345**, 244.

1-Phenylcrotonyl Alcohol.
See Propenylphenylcarbinol.

Phenylcyanamide (*Cyananilide, phenylcarbamic nitrile*, N-*cyanoaniline*)

$C_6H_5·NH·CN$

$C_7H_6N_2$ MW, 118

Cryst. from H_2O or Et_2O. M.p. 47°. Sol. EtOH, Et_2O. Spar. sol. H_2O. Sol. KOH.Aq.

Berger, *Monatsh.*, 1884, **5**, 219.

Phenyl cyanide.
See Benzonitrile.

Phenylcyanoacetic Acid (α-*Cyanophenylacetic acid, phenylmalonic mononitrile*)

$C_6H_5·CH(CN)·COOH$

$C_9H_7O_2N$ MW, 161

Cryst. from Et_2O–ligroin. M.p. 92°. Sol. H_2O, EtOH, Et_2O, hot C_6H_6. Insol. ligroin. At 150–60° ⟶ benzyl cyanide.

Et ester: $C_{11}H_{11}O_2N$. MW, 189. Oil. B.p. 275° slight decomp., 165·5°/20 mm. Misc. with org. solvents. Insol. H_2O.

Amide: $C_9H_8ON_2$. MW, 160. Cryst. from EtOH. M.p. 147°. Sol. hot EtOH. Insol. H_2O.

Methylamide: $C_{10}H_{10}ON_2$. MW, 174. M.p. 102°.

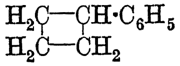
Anilide: needles from EtOH. M.p. 136°. Spar. sol. Et_2O. Insol. H_2O.

o-*Toluidide*: m.p. 139°.
m-*Toluidide*: m.p. 131°.
p-*Toluidide*: needles from EtOH. M.p. 139°.

Merck, D.R.P., 606,349, (*Chem. Zentr.*, 1935, I, 2048).
Heseler, *Am. Chem. J.*, 1904, **32**, 122.

2-Phenyl-3-cyanocinchoninic Acid.
See under 2-Phenylquinoline-3 : 4-dicarboxylic Acid.

Phenylcyclobutane (*Cyclobutylbenzene*)

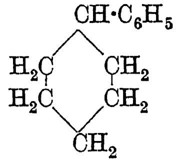


$C_{10}H_{12}$ MW, 132

B.p. 190–1°/755 mm., 101–2°/41 mm. D_4^{20} 0·9378. n_D^{20} 1·5277.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

Phenylcyclohexane (*Cyclohexylbenzene, hexahydrodiphenyl*)



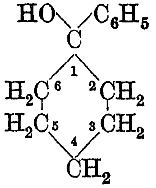
$C_{12}H_{16}$ MW, 160

Plates. M.p. 7–8°. B.p. 235–6°, 156°/80 mm., 127–8°/30 mm., 106°/12 mm. D_4^{20} 0·9502. n_D^{20} 1·5329. Stable to cold $KMnO_4$.

Meerwein, *Ann.*, 1919, **419**, 171.
Bedos, *Compt. rend.*, 1923, **177**, 111.
Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.
Neunhoffer, *J. prakt. Chem.*, 1932, **133**, 95.
Truffault, *Bull. soc. chim.*, 1934, **1**, 391.
Tsukervanik, Sidorova, *J. Gen. Chem. U.S.S.R.*, 1937, **7**, 641, (*Chem. Abstracts*, 1937, **31**, 5780).

Phenylcyclohexane-carboxylic Acid.
See Phenylhexahydrobenzoic Acid.

1-Phenylcyclohexanol (1-*Hydroxyhexahydrodiphenyl*, 1-*phenylhexahydrophenol*)



$C_{12}H_{16}O$ MW, 176

Prisms from ligroin. M.p. 63–63·5°. B.p. 156·5–158·5°/28 mm., 141–4°/14 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, ligroin. Insol. H_2O.

Auwers, Treppmann, *Ber.*, 1915, **48**, 1216.
Sabatier, Mailhe, *Compt. rend.*, 1904, **138**, 1322.
Kurssanow, *Chem. Zentr.*, 1907, I, 1744.

2-Phenylcyclohexanol (*2-Hydroxyhexahydrodiphenyl, 2-phenylhexahydrophenol*).

Exists in *cis* and *trans* forms. *Cis-*: m.p. 56–7°. B.p. 140°/16 mm. *Trans-*: m.p. 56–7°. B.p. 152–5°/16 mm.

Me ether: b.p. 165–7°/3 mm. D_{25}^{25} 0·9791. n_D^{25} 1·5082.

Et ether: b.p. 110–13°/4 mm. D_{25}^{25} 0·9653. n_D^{25} 1·5065.

3 : 5-*Dinitrobenzoyl*: cryst. from EtOH. M.p. 121–121·5°.

Phenylurethane: *cis-*: m.p. 117–18°. *Trans-*: m.p. 137–138·5°.

Phthalate: m.p. 185–6°.

Bedos, *Bull. soc. chim.*, 1926, **39**, 292.
Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 75.

3-Phenylcyclohexanol (*3-Hydroxyhexahydrodiphenyl, 3-phenylhexahydrophenol*).

Needles from pet. ether. M.p. 81° (79·5–80·5°). Sol. most org. solvents. Volatile in steam. Odour resembles geranium.

Acetyl: plates from EtOH. M.p. 43–4°. B.p. 300°.

Benzoyl: prisms from EtOH. M.p. 68°.

o-*Nitrobenzoyl*: needles from EtOH. M.p. 70°.

Boyd, Clifford, Probert, *J. Chem. Soc.*, 1920, **117**, 1383.
Crossley, Renouf, *J. Chem. Soc.*, 1915, **107**, 608.

4-Phenylcyclohexanol (*4-Hydroxyhexahydrodiphenyl, 4-phenylhexahydrophenol*).

Cis:
M.p. 76–7°.
Phenylurethane: m.p. 140·5°.

Trans:
Needles from C_6H_6. M.p. 117–18°. B.p. 155–8°/13 mm. Very sol. Et_2O. Mod. sol. ligroin. Spar. sol. H_2O.

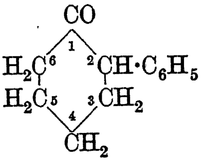
Benzoyl: m.p. 104·5–105·5°.

p-*Toluenesulphonyl*: m.p. 98–9°.

Phenylurethane: m.p. 140–1°.

Kurssanow, *Ann.*, 1901, **318**, 325; *Chem. Zentr.*, 1923, III, 1075.
Wuyts, *Chem. Zentr.*, 1912, II, 1006.

2-Phenylcyclohexanone



$C_{12}H_{14}O$ MW, 174

Cryst. from pet. ether or EtOH. M.p. 63° (50–3°). B.p. 180°/16 mm. (155–60°/16 mm., 136–7°/16 mm.). Spar. sol. H_2O. Does not form bisulphite comp.

Oxime: m.p. 169°.

Semicarbazone: m.p. 190°.

Le Brazidec, *Compt. rend.*, 1914, **159**, 775.
v. Braun, Gruber, Kirschbaum, *Ber.*, 1922, **55**, 3670.
Levy, Stiras, *Bull. soc. chim.*, 1931, **49**, 1830.
Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 76.
Sherwood, Short, Woodcock, *ibid.*, 323.
Newman, Farbman, *J. Am. Chem. Soc.*, 1944, **66**, 1550.

3-Phenylcyclohexanone.

Oil. B.p. 287–8°/736 mm., 169–169·5°/18 mm. Alc. $H_2SO_4 \longrightarrow$ reddish-yellow sol. with green fluor.

Oxime: plates from EtOH. M.p. 128–9°. Sol. most org. solvents. Mod. sol. Et_2O. Insol. pet. ether.

Semicarbazone: prisms from EtOH. M.p. 167°. Sol. MeOH, EtOH, Me_2CO, $CHCl_3$. Mod. sol. Et_2O.

2 : 4-*Dinitrophenylhydrazone*: m.p. 183–6°.

Boyd, Clifford, Probert, *J. Chem. Soc.*, 1920, **117**, 1383.

4-Phenylcyclohexanone.

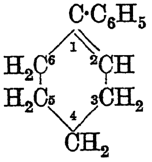
Cryst. from pet. ether. M.p. 78°. B.p. 158–60°/12 mm.

Oxime: m.p. 110–11°.

Semicarbazone: cryst. from EtOH. M.p. 229° decomp.

v. Braun, Weissbach, *Ber.*, 1931, **64**, 1788.

1-Phenylcyclohexene (3 : 4 : 5 : 6-*Tetrahydrodiphenyl*)



$C_{12}H_{14}$ MW, 158

Mobile liq. B.p. 250°, 133°/20 mm., 125–6°/14 mm. $D_4^{14·9}$ 0·9930, D_4^{20} 0·9931, $D_4^{25·2}$ 0·9871. n_D^{20} 1·5718 (1·5670), $n_D^{25·2}$ 1·5669. $KMnO_4 \longrightarrow$ 4-benzoylvaleric acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, **62**, 1160.
Sabatier, Mailhe, *Compt. rend.*, 1904, **138**, 1323.

3-Phenylcyclohexene (1 : 2 : 3 : 4-*Tetrahydrodiphenyl*).

B.p. 115–17°/16 mm. D_4^{20} 0·9800. n_D^{20} 1·5530. Reflux with 5% $HNO_3 \longrightarrow$ 2-phenyladipic acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, **62**, 1160.

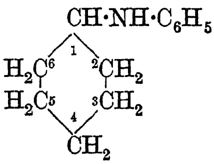
4-Phenylcyclohexene (1 : 2 : 5 : 6-*Tetrahydrodiphenyl*).

B.p. 235°/760 mm., 88–90°/16 mm. D_4^{20}

0·9715. n_D^{20} 1·5420. $KnMO_4$ ⟶ 2-phenyl-adipic acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, **62**, 2243.

N-Phenylcyclohexylamine (*Hexahydrodiphenylamine*, N-*cyclohexylaniline*)



$C_{12}H_{17}N$ MW, 175

M.p. 14°. B.p. 191–2°/73 mm., 180·5–182·5°/66 mm., 134–5°/6 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

B,HCl: needles. M.p. 230·5° (204–5°). Sol. EtOH. Spar. sol. Et_2O.

B_2,H_2SO_4: needles. M.p. 226–227·5°.

B,HBr: needles. M.p. 184°.

B,HI: needles. M.p. 176°.

B,HNO_3: m.p. 173°.

Acetate: needles. M.p. 154–155·5°.

N-*Acetyl*: m.p. 69–70°. Sol. EtOH. Insol. H_2O.

N-*Benzoyl*: m.p. 161–2°.

N-p-*Toluenesulphonyl*: m.p. 141–2°.

B_2,H_2PtCl_6: m.p. 177°.

Picrate: yellow prisms. M.p. 164–5°.

Kurssanow, *Chem. Zentr.*, 1907, I, 1744.
Guyot, Fournier, *Bull. soc. chim.*, 1930, **47**, 203.
Skita, Keil, *Ber.*, 1928, **61**, 1682.
Bucherer, Fischbeck, *J. prakt. Chem.*, 1934, **140**, 69.
Hickinbottom, *J. Chem. Soc.*, 1932, 2646.
Hiers, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 1102.
I.G., D.R.P., 483,205, (*Chem. Zentr.*, 1929, II, 2938).
Forge, *Ann. chim.*, 1921, **15**, 291.

2-Phenylcyclohexylamine (1-*Amino*-ac-*hexahydrodiphenyl*).

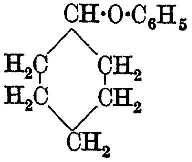
M.p. 58–9°. B.p. 110–12°/3 mm.

B,HCl: m.p. 253°.

N-*Benzoyl*: m.p. 182°.

Nightingale, Tweedie, *J. Am. Chem. Soc.*, 1944, **66**, 1968.

Phenyl cyclohexyl Ether (*Hexahydrodiphenyl ether*, *phenoxycyclohexane*)

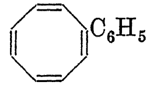


$C_{12}H_{16}O$ MW, 176

B.p. 260–2°, 140°/21·5 mm., 128–128·5°/15 mm. D_4^0 1·0241, D_4^{20} 1·0077. n_D^{22} 1·520.

Kurssanow, *J. Russ. Phy.-Chem. Soc.*, 1916, **48**, 1172.
Scranth, Quasebath, *Ber.*, 1924, **57**, 854.
v. Duzee, Adkins, *J. Am. Chem. Soc.*, 1935, **57**, 147.
Lefebvre, Levas, *Compt. rend.*, 1945, **221**, 301.
Levas, *Ann. chim.*, 1948, [12], **3**, 145.

Phenylcyclo-octatetraene



$C_{14}H_{12}$ MW, 180

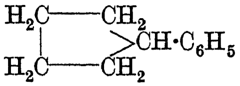
Yellow oil. B.p. 101–3°/0·4–0·55 mm. n_D^{25} 1·6121.

$AgNO_3$ *add. comp.*: cryst. M.p. 143·5° decomp.

Maleic anhydride add. comp.: cryst. M.p. 172–4°.

Cope, Van Orden, *J. Am. Chem. Soc.*, 1952, **74**, 175.
Cope, Campbell, *ibid.*, 179.

Phenylcyclopentane (*Cyclopentylbenzene*)

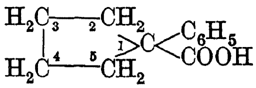


$C_{11}H_{14}$ MW, 146

B.p. 213–15°, 116–17°/37 mm. D_4^{20} 0·9502, D_4^{25} 0·9432. n_D^{20} 1·5309.

Zelinski, *Ber.*, 1925, **58**, 2755.
v. Braun, Kühn, *Ber.*, 1927, **60**, 2561.
Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.
Dupont, *Chem. Zentr.*, 1936, II, 613.
Cagniant, Debuzarche, Chatelus, *Compt. rend.*, 1947, **224**, 1064.

1-Phenylcyclopentane-1-carboxylic Acid



$C_{12}H_{14}O_2$ MW, 190

M.p. 158–9°.

Amide: $C_{12}H_{15}ON$. MW, 189. Cryst. from C_6H_6. M.p. 157–8°.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

2-Phenylcyclopentane-1-carboxylic Acid. Thick oil. B.p. 190–2°/13 mm.

Chloride: $C_{12}H_{13}OCl$. MW, 208·5. B.p. 150–3°/12 mm.

Anilide: cryst. from Et_2O. M.p. 93–5°.

v. Braun, Kühn, *Ber.*, 1927, **60**, 2560.

3-Phenylcyclopentane-1-carboxylic Acid. B.p. 196–8°/5 mm.

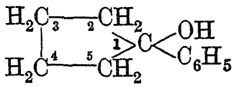
Chloride: b.p. 159–62°/15 mm.

Amide: $C_{12}H_{15}ON$. MW, 189. M.p. 149°.

Anilide: m.p. 107°.

Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 268.

1-Phenylcyclopentanol



$C_{11}H_{14}O$ MW, 162

B.p. 132–3°/18 mm., 121·0–121·1°/6 mm. D_4^{20} 1·0530. n_D^{20} 1·5479.

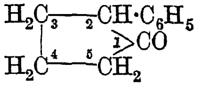
Zelinski, *Ber.*, 1925, **58**, 2755.
Dupont, Chavanne, *Bull. soc. chim. Belg.*, 1933, **42**, 537.

3-Phenylcyclopentanol.

B.p. 155–6°/10 mm.
Acetyl: thick oil. B.p. 154°/12 mm.

Borsche, Menz, *Ber.*, 1908, **41**, 203.

2-Phenylcyclopentanone



$C_{11}H_{12}O$ MW, 160

M.p. 126–7°.
Oxime: m.p. 146° decomp.
Semicarbazone: m.p. 228° decomp.

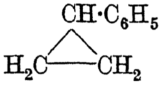
Mitchovitch, *Compt. rend.*, 1935, **200**, 1601.

3-Phenylcyclopentanone.

B.p. 154–5°/10 mm.
Semicarbazone: leaflets from EtOH. M.p. 181° decomp.

Borsche, Menz, *Ber.*, 1908, **41**, 204.
Wilson Baker, Leeds, *J. Chem. Soc.*, 1948, 974.

Phenylcyclopropane (*Cyclopropylbenzene*)

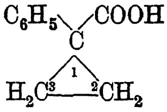


C_9H_{10} MW, 118

B.p. 173·6°/758 mm., 79–80°/37 mm. D_4^{20} 0·9317. n_D^{20} 1·5285. 60% H_2SO_4 ⟶ dimer.
Dimer: thick oil. B.p. 330–2°/750 mm. D_0^{17} 1·002. n_D^{17} 1·5710.

Kishner, *Chem. Zentr.*, 1913, II, 2129.
Lespieau, *Compt. rend.*, 1930, **190**, 1129.
Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

1-Phenylcyclopropane-1-carboxylic Acid



$C_{10}H_{10}O_2$ MW, 162

M.p. 86–7°.
Amide: $C_{10}H_{11}ON$. MW, 161. M.p. 100–1°.
Nitrile: $C_{10}H_9N$. MW, 143. B.p. 250–3°/751 mm., 98–100°/1 mm. D_4^{20} 1·0156. n_D^{20} 1·3676.

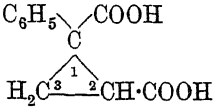
Knowles, Cloke, *J. Am. Chem. Soc.*, 1932, **54**, 2028; *Chem. Abstracts*, 1935, **29**, 2156.
Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

2 - Phenylcyclopropane - 1 - carboxylic Acid.

Needles from H_2O. M.p. 105°.
Et ester: $C_{12}H_{14}O_2$. MW, 190. Needles from ligroin. M.p. 39°. B.p. 144–8°/15 mm.
Amide: leaflets from hot H_2O. M.p. 187–8°.

Buchner, Geronimus, *Ber.*, 1903, **36**, 3783.

1-Phenylcyclopropane-1 : 2-dicarboxylic Acid



$C_{11}H_{10}O_4$ MW, 206

Free acid unstable.
Anhydride: $C_{22}H_{18}O_7$. MW, 394. Needles from EtOH.Aq. M.p. 99°. Sol. EtOH, Et_2O, C_6H_6. Sol. hot H_2O with acid reaction.

Ruhemann, *J. Chem. Soc.*, 1902, **81**, 1215.

3-Phenylcyclopropane-1 : 2-dicarboxylic Acid.

Cis:
Prisms from EtOH. M.p. 175°. Sol. EtOH, Et_2O. Less sol. H_2O. Heat in vacuo ⟶ anhydride.
Di-Me ester: $C_{13}H_{14}O_4$. MW, 234. Needles from ligroin. M.p. 63°. B.p. 200–14°/20 mm.
Di-Et ester: $C_{15}H_{18}O_4$. MW, 262. Viscous oil. B.p. 256–7°/120 mm.
Anhydride: cryst. from boiling Et_2O. M.p. 134°. B.p. 282°/190 mm.

Trans:
Needles from H_2O. M.p. 121°.

Buchner, Dessauer, *Ber.*, 1892, **25**, 1147.
Buchner, Perkel, *Ber.*, 1903, **36**, 3777.
Buchner, *Ber.*, 1888, **21**, 2645.
Haerdi, Thorpe, *J. Chem. Soc.*, 1925, 1243.

S-Phenylcysteine (β-*Phenylmercaptoalanine*)

$C_6H_5{\cdot}S{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$

$C_9H_{11}O_2NS$ MW, 197

Plates from hot H_2O. Decomp. at 170–2°. $[\alpha]_D^{25}$ + 11° in 0·1*N* NaOH.

Zbarsky, Young, *J. Biol. Chem.*, 1943, **151**, 211.

β-Phenylcysteine (2-*Mercapto*-1-*amino*-2-*phenylpropionic acid*)

$C_6H_5{\cdot}CH(SH){\cdot}CH(NH_2){\cdot}COOH$

$C_9H_{11}O_2NS$ MW, 197

B,HCl: plates from isopropyl alcohol-pet. ether. M.p. 202–3° decomp. Sol. EtOH. Mod. sol. H_2O, Me_2CO, $CHCl_3$, isopropyl alcohol, *tert.*-butyl alcohol.

Cook, Harris, Heilbron, *J. Chem. Soc.*, 1948, 1060.

N-Phenyldiacetamide.
See Diacetanilide.

Phenyl-diaminoditolyl-methane.
See Diaminodimethyltriphenylmethane.

N-Phenyldibenzamide.
See Dibenzanilide.

2-Phenyl-1 : 3-dibenzoylpropane.
See Benzylidene-diacetophenone.

Phenyldibenzylamine.
See Dibenzylaniline.

Phenyldibromoarsine (*Dibromo-phenylarsine, phenylarsen-dibromide*)

$C_6H_5AsBr_2$

$C_6H_5Br_2As$ MW, 312

B.p. 285° decomp. D^{15} 2·0983.

La Coste, Michaelis, *Ann.*, 1880, **201**, 203.
Winmill, *J. Chem. Soc.*, 1912, **101**, 723.
Roeder, Blasi, *Ber.*, 1914, **47**, 2752.

Phenyldichloroacetic Acid.
See α : α-Dichlorophenylacetic Acid.

Phenyldichloroarsine (*Dichloro-phenylarsine, phenylarsen-dichloride*)

$C_6H_5AsCl_2$

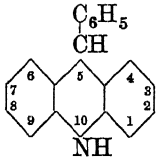
$C_6H_5Cl_2As$ MW, 223

B.p. 252–5°.

La Coste, Michaelis, *Ann.*, 1880, **201**, 193.
Winmill, *J. Chem. Soc.*, 1912, **101**, 720.
Hanby, Waters, *J. Chem. Soc.*, 1946, 1029.
Barker *et al.*, *J. Soc. Chem. Ind.*, 1949, **68**, 290.

Phenyl ω-dichlorobenzyl Ketone.
See αα-Dichlorodeoxybenzoin.

5-Phenyl-5 : 10-dihydroacridine



$C_{19}H_{15}N$ MW, 257

Needles from propyl alcohol or C_6H_6. M.p. 170° (163°). Mod. sol. Et_2O, hot EtOH.

N-*Me*: $C_{20}H_{17}N$. MW, 271. Needles. M.p. 104°.

N-*Acetyl*: cryst. from C_6H_6–ligroin. M.p. 128°.

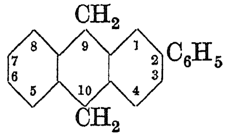
Bernthsen, *Ann.*, 1884, **224**, 25.
Bergmann, Blum-Bergmann, v. Christiani, *Ann.*, 1930, **483**, 85.

10-Phenyl-5 : 10-dihydroacridine (N-*Phenyldihydroacridine*).

Needles or prisms from AcOH. M.p. 119°. Very sol. Et_2O, C_6H_6. Sol. EtOH. Insol. H_2O. Alc. sol. shows blue fluor.

Ullmann, Maag, *Ber.*, 1907, **40**, 2518.

2-Phenyl-9 : 10-dihydroanthracene



$C_{20}H_{16}$ MW, 256

Yellowish cryst. from AcOH. M.p. 93–6°. Sol. $CHCl_3$, Me_2CO, CCl_4, CS_2. Mod. sol. Et_2O, AcOH. CrO_3 in boiling AcOH ⟶ 2-phenylanthraquinone.

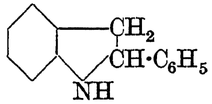
Scholl, Neovius, *Ber.*, 1911, **44**, 1081.

9-Phenyl-9 : 10-dihydroanthracene.
Needles from MeOH. M.p. 91–91·5°.

Haack, *Ber.*, 1929, **62**, 1783.

2-Phenyl-2 : 3-dihydrobenz-γ-pyrone.
See Flavanone.

2-Phenyl-2 : 3-dihydroindole (*2-Phenylindoline*)



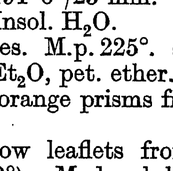
$C_{14}H_{13}N$ MW, 195

Cryst. from ligroin. M.p. 46°. B.p. 184–6°/10 mm. n_D^{25} 1·6186. Sol. dil. min. acids. Volatile in steam.

B_2,H_2PtCl_6: yellowish-red needles. M.p. 191° decomp. Insol. EtOH.

Pictet, *Ber.*, 1886, **19**, 1065.
Stoermer, *Ber.*, 1898, **31**, 2540.
Adkins, Coonradt, *J. Am. Chem. Soc.*, 1941, **63**, 1563.

1-Phenyl-3 : 4-dihydroisoquinoline



$C_{15}H_{13}N$ MW, 207

Prisms from pet. ether. M.p. 73–4°. B.p. 320°/718 mm., 194–6°/23 mm. Spar. sol. usual org. solvents. Insol. H_2O.

B,HCl: needles. M.p. 225°. Sol. H_2O, EtOH, $CHCl_3$. Insol. Et_2O, pet. ether.

B_2,H_2PtCl_6: orange prisms from H_2O. M.p. 230–3°.

Picrate: yellow leaflets from $CHCl_3$–Et_2O. M.p. 173–5° (163°). Mod. sol. hot H_2O.

Pictet, Kay, *Ber.*, 1909, **42**, 1975.
Späth, Berger, Kuntara, *Ber.*, 1930, **63**, 139.
Dey, Ramanathan, *Chem. Abstracts*, 1949, **43**, 5025.

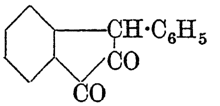
3-Phenyl-3 : 4-dihydroquinazoline.
See Orexine.

Phenyl 2 : 4-dihydroxyphenacyl Ether.
See under Fisetol.

Phenyl 3 : 4-dihydroxystyryl Ketone.
See 3 : 4-Dihydroxychalkone.

Phenyl dihydroxytolyl Ketone.
See Dihydroxy-methylbenzophenone.

3-Phenyl-1 : 2-diketohydrindene (*3-Phenylindandione*-1 : 2)

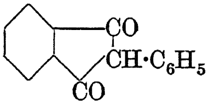


$C_{15}H_{10}O_2$ MW, 222

Needles from ligroin. M.p. 137–8°.
Disemicarbazone: yellow needles from EtOH. M.p. 252° decomp.

Pfeiffer, Waal, *Ann.*, 1935, **520**, 192.

2-Phenyl-1 : 3-diketohydrindene (*2-Phenylindandione*-1 : 3)

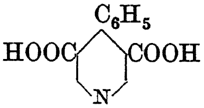


$C_{15}H_{10}O_2$ MW, 222

Leaflets from EtOH. M.p. 147–8° (145°). Sol. usual org. solvents. Insol. H_2O. Sol. conc. H_2SO_4 with blue col. Sol. alkalis with deep red col.
Dioxime: needles from EtOH. M.p. 193–6°.

Nathanson, *Ber.*, 1893, **26**, 2576.
Hantzsch, Gajewski, *Ann.*, 1912, **392**, 303.

4-Phenyldinicotinic Acid (*4-Phenylpyridine-3 : 5-dicarboxylic acid*)



$C_{13}H_9O_4N$ MW, 243

Greenish-yellow plates + $\frac{1}{2}H_2O$. M.p. 229–30°, anhyd. 245–6° decomp.

Weber, *Ann.*, 1887, **241**, 13.

***N*-Phenyl-dinitrobenzylamine.**
See Dinitrobenzylaniline.

Phenyldiphenacylmethane.
See Benzylidene-diacetophenone.

Phenyl-diphenyl.
See Diphenylbenzene.

Phenyldiphenylenemethane.
See 9-Phenylfluorene.

Phenyldiphenylylmethane.
See *p*-Benzyldiphenyl.

Phenyldithiocarbamic Acid (*Dithiocarbanilic acid*)

$C_6H_5·NH·CSSH$

$C_7H_7NS_2$ MW, 169

NH_4 salt: yellow prisms. M.p. 108° decomp. Sol. 4 parts H_2O at 35°. Spar. sol. EtOH.
Me ester: phenyldithiourethylan. $C_8H_9NS_2$. MW, 183. Leaflets from EtOH.Aq. M.p. 95–6° (87–8°).
Et ester: *see* Phenyldithiourethane.
Propyl ester: $C_{10}H_{13}NS_2$. MW, 211. Cryst. from pet. ether. M.p. 66–7°.
Allyl ester: $C_{10}H_{11}NS_2$. MW, 209. M.p. 42°.
Isoamyl ester: $C_{12}H_{17}NS_2$. MW, 239. Plates. M.p. 71°.
Phenyl ester: $C_{13}H_{11}NS_2$. MW, 245. Cryst. from EtOH. M.p. 104–6° decomp.
Benzyl ester: $C_{14}H_{13}NS_2$. MW, 259. Cryst. from EtOH. M.p. 84–5°.

Freund, Bachrach, *Ann.*, 1895, **285**, 199.
v. Braun, *Ber.*, 1902, **35**, 3384.
Roshdestwenski, *Chem. Zentr.*, 1910, I, 910.

Phenyldithiourethane (*Dithiocarbanilic acid ethyl ester*)

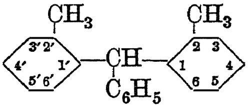
$C_6H_5·NH·CS·SC_2H_5$

$C_9H_{11}NS_2$ MW, 197

Plates from EtOH. M.p. 60–1°. Very sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

Will, *Ber.*, 1882, **15**, 1305.
Losanitsch, *Ber.*, 1891, **24**, 3025.

Phenyldi-*o*-tolylmethane (*2 : 2′-Dimethyltriphenylmethane*)



$C_{21}H_{20}$ MW, 272

Cryst. from AcOH. M.p. 104°. B.p. 180–5°/12 mm.

Weiss, Reichel, *Monatsh.*, 1929, **53**, 197.
Bredereck *et al.*, *Ber.*, 1939, **72**, 1426.

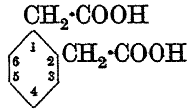
Phenyldi-*p*-tolylmethane (*4 : 4′-Dimethyltriphenylmethane*).

Needles from MeOH. M.p. 55–6° (52°). Very sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Less sol. EtOH, AcOH, ligroin.

Kliegl, *Ber.*, 1905, **38**, 85.
Guyot, Kovache, *Compt. rend.*, 1912, **154**, 122.
Vorländer, *Ber.*, 1911, **44**, 2470.

Phenyldodecane.
See Dodecylbenzene.

***o*-Phenylenediacetic Acid** (*o-Xylene-ω : ω′-dicarboxylic acid, benzene-o-diacetic acid*)



$C_{10}H_{10}O_4$ MW, 194

Needles from H_2O or Et_2O. M.p. 150° (148·5–149°). Sol. EtOH, Et_2O, hot H_2O. $k = 1·111 \times 10^{-4}$ at 25°.

Di-Et ester: $C_{14}H_{18}O_4$. MW, 250. Oil. B.p. 185°/15 mm.

Diamide: $C_{10}H_{12}O_2N_2$. MW, 192. Needles from H_2O. M.p. 198°. Spar. sol. EtOH.

Dinitrile: *o*-xylylene dicyanide, ω : ω′-dicyano-*o*-xylene. $C_{10}H_8N_2$. MW, 156. Exists in two forms. (i) Labile. Prisms or needles from MeOH. M.p. 18°. (ii) Stable. Prisms from EtOH. M.p. 60°. Sol. EtOH, Et_2O.

Moore, Thorpe, *J. Chem. Soc.*, 1908, **93**, 175.
Baeyer, Pape, *Ber.*, 1884, **17**, 447.
v. Braun, Kruber, Danziger, *Ber.*, 1916, **49**, 2648.
Morton, Little, Strong, *J. Am. Chem. Soc.*, 1943, **65**, 1345.

m-Phenylenediacetic Acid (m-*Xylene-ω : ω′-dicarboxylic acid, benzene-*m*-diacetic acid*).

Needles from H_2O. M.p. 170°.

Di-Me ester: $C_{12}H_{14}O_4$. MW, 222. B.p. 298–300°, 185–7°/15 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, CS_2.

Di-Et ester: b.p. 188–9°/12 mm.

Dinitrile: *m*-xylylene dicyanide, ω : ω′-dicyano-*m*-xylene. Cryst. M.p. 28–9°. B.p. 305–10°/300 mm. part. decomp., 170°/20–30 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O, ligroin.

Kipping, Oddo, *J. Chem. Soc.*, 1888, **53**, 41.
Oddo, *Gazz. chim. ital.*, 1893, **23**, ii, 337.
Titley, *J. Chem. Soc.*, 1928, 2579.
Morton, Little, Strong, *J. Am. Chem. Soc.*, 1943, **65**, 1345.

p-Phenylenediacetic Acid (p-*Xylene-ω : ω′-dicarboxylic acid, benzene-*p*-diacetic acid*).

Needles from H_2O. M.p. 244° (236°). Sol. H_2O, EtOH, Et_2O. Spar. sol. $CHCl_3$, CS_2, pet. ether.

Di-Me ester: leaflets. M.p. 56·5–57°. B.p. 189–90°/15 mm.

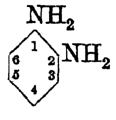
Di-Et ester: m.p. 59°.

Diamide: leaflets or needles from H_2O. Does not melt below 290°. Spar. sol. usual solvents.

Dinitrile: *p*-xylylene dicyanide, ω : ω′-dicyano-*p*-xylene. Needles from H_2O, prisms from Et_2O. M.p. 98°. Sol. EtOH, Et_2O, $CHCl_3$. Mod. sol. hot H_2O.

Titley, *J. Chem. Soc.*, 1928, 2579.
Zincke, Klippert, *Ber.*, 1876, **9**, 1767.
Kipping, *J. Chem. Soc.*, 1888, **53**, 44.
Morton, Little, Strong, *J. Am. Chem. Soc.*, 1943, **65**, 1345.

o-Phenylenediamine (1 : 2-*Diaminobenzene*)



$C_6H_8N_2$ MW, 108

Leaflets from H_2O, plates from $CHCl_3$. M.p. 102–3°. B.p. 256–8°/760 mm. Very sol. EtOH, Et_2O, $CHCl_3$. Sol. hot H_2O. $k = 3{\cdot}3 \times 10^{-10}$ at 25°.

N-*Me*: *see N*-Methyl-*o*-phenylenediamine.

N-*Di-Me*: *see o*-Aminodimethylaniline.

N : N′-*Di-Me*: *see sym.*-Dimethyl-*o*-phenylenediamine.

N-*Et*: *see N*-Ethyl-*o*-phenylenediamine.

N-*Di-Et*: *see o*-Aminodiethylaniline.

N-*Phenyl*: *see o*-Aminodiphenylamine.

N-*Diphenyl*: *see o*-Aminotriphenylamine.

N : N′-*Diphenyl*: *see sym.*-Diphenyl-*o*-phenylenediamine.

N : N′-*Tetra-Me*: $C_{10}H_{16}N_2$. MW, 164. Oil. B.p. 215–18°/735 mm. Hot $FeCl_3 \longrightarrow$ red col. *B,2HCl*: prisms. M.p. 180°.

N-p-*Tolyl*: *see* 2′-Amino-4-methyldiphenylamine.

N-*Dibenzyl*: $C_{20}H_{20}N_2$. MW, 288. Oil. N′-*Acetyl*: needles from EtOH. M.p. 121–2°. N′-*Benzoyl*: needles from AcOH. M.p. 156°.

N : N′-*Dibenzyl*: cryst. from C_6H_6–pet. ether. M.p. 21°. *B,HCl*: needles from EtOH.Aq. + HCl. M.p. 149°.

N-*Benzylidene*: yellow cryst. from pet. ether. M.p. 60–1°. Sol. EtOH, Et_2O, ligroin. N′-*Acetyl*: golden-yellow leaflets. M.p. 125°.

N : N′-*Dibenzylidene*: prisms from ligroin. M.p. 106°.

N-*Acetyl*: *see o*-Aminoacetanilide.

N : N′-*Diacetyl*: needles from H_2O. M.p. 185–6°. Sol. boiling H_2O, EtOH, $CHCl_2$, Me_2CO, AcOH. Spar. sol. Et_2O, C_6H_6, ligroin.

N-*Benzoyl*: cryst. from H_2O. M.p. 140°. Sol. EtOH.

N : N′-*Dibenzoyl*: prisms from AcOH. M.p. 301°. Spar. sol. most org. solvents. N-*Formyl*: needles from $CHCl_3$-ligroin. M.p. 157°. Sol. Me_2CO, AcOEt, hot C_6H_6. Spar. sol. Et_2O. N-*Propionyl*: cryst. from $CHCl_3$–ligroin. M.p. 124°.

N : N′-*Di-o-nitrobenzoyl*: pale yellow needles from AcOH. M.p. 265°. Sol. hot EtOH.

N : N′-*Di*-m-*nitrobenzoyl*: needles from AcOH. M.p. 240°. Mod. sol. hot EtOH, AcOH. Spar. sol. C_6H_6.

N-p-*Nitrobenzoyl*: yellow needles from H_2O. M.p. 200°. Sol. EtOH, AcOH. Spar. sol. H_2O. Insol. C_6H_6.

N : N′-*Di*-p-*nitrobenzoyl*: prisms from AcOH. M.p. 267°. Sol. hot EtOH. Insol. Et_2O, C_6H_6.

N-*Picryl*: red cryst. from xylene. M.p. 177–8° decomp. Sol. Me_2CO, xylene, $PhNO_2$. Spar. sol. EtOH, Et_2O.

N-p-*Toluenesulphonyl*: m.p. 259–260·5°.

N-*Carbethoxyl*: *see o*-Aminophenylurethane.

Hinsberg, König, *Ber.*, 1895, **28**, 2947.
Martin, *Organic Syntheses*, 1939, XIX, 70.
Hazlet, Dornfeld, *J. Am. Chem. Soc.*, 1944, **66**, 1781.

***m*-Phenylenediamine** (1 : 3-*Diaminobenzene*).

Rhombic cryst. from EtOH. M.p. 63–4°. B.p. 282–4°/760 mm. D_{10}^{10} 1·1421, $D_4^{57\cdot7}$ 1·10696. $n_D^{57\cdot7}$ 1·63390. Very sol. H_2O, EtOH. Less sol. Et_2O.

N-*Me* : *see* *N*-Methyl-*m*-phenylenediamine.

N-*Di-Me* : *see* *m*-Aminodimethylaniline.

N : N′-*Di-Me* : *see* *sym.*-Dimethyl-*m*-phenylenediamine.

N-*Et* : *see* *N*-Ethyl-*m*-phenylenediamine.

N-*Di-Et* : *see* *m*-Aminodiethylaniline.

N-*Phenyl* : *see* *m*-Aminodiphenylamine.

N-*Diphenyl* : *see* *m*-Aminotriphenylamine.

N : N′-*Diphenyl* : *see* *sym.*-Diphenyl-*m*-phenylenediamine.

N : N′-*Tri-Me* : $C_9H_{14}N_2$. MW, 150. B.p. 270° (280°). Sol. EtOH, C_6H_6. *Methiodide* : cryst. + H_2O. M.p. 192°. Sol. H_2O, EtOH. Insol. Et_2O. *Acetyl* : cryst. from EtOH. M.p. 68°. B.p. 280°.

N : N′-*Tetra-Me* : $C_{10}H_{16}N_2$. MW, 164. Oil. F.p. — 2°. B.p. 266–7°/748 mm. $D^{15\cdot8}$ 0·9849. *Methiodide* : cryst. + $1H_2O$ from H_2O. M.p. 192°.

N : N′-*Tetraphenyl* : $C_{30}H_{24}N_2$. MW, 412. Needles from Me_2CO. M.p. 137·5–138°. Sol. Me_2CO, C_6H_6. Less sol. AcOH. Sol. 800 parts MeOH at 15°.

N : N′-*Di*-p-*tolyl* : *see* Di-*p*-tolyl-*m*-phenylenediamine.

N-1-*Naphthyl* : *m*-aminophenyl-1-naphthylamine. $C_{16}H_{14}N_2$. MW, 234. Prisms. M.p. 94–5°. B.p. 275–80°/12 mm. Very sol. EtOH, C_6H_6. Sol. hot Et_2O. Mod. sol. hot H_2O.

N-2-*Naphthyl* : *m*-aminophenyl-2-naphthylamine. $C_{16}H_{14}N_2$. MW, 234. Needles from EtOH. M.p. 128°. B.p. 320°/40 mm. Very sol. Et_2O, $CHCl_3$. *B,2HCl* : m.p. 210°. *Picrate* : leaflets. M.p. 180° decomp. N′-*Acetyl* : needles from EtOH. M.p. 135°. N : N′-*Diacetyl* : m.p. 147–8°. N′-*Benzoyl* : m.p. 173°. N : N′-*Dibenzoyl* : m.p. 213°.

Dinaphthyl : *see* Dinaphthyl-*m*-phenylenediamine.

N-*Dibenzyl* : $C_{20}H_{20}N_2$. MW, 288. Oil. N′-*Acetyl* : needles from EtOH. M.p. 143–4°. N′-*Benzoyl* : needles from AcOH. M.p. 171–2°.

N : N′-*Tetrabenzyl* : amorph. M.p. 80–1°.

Dibenzylidene : yellowish needles from Et_2O. M.p. 104–5°.

N : N′-*Diformyl* : cryst. from EtOH. M.p. 155°. Sol. hot H_2O, EtOH.

N-*Acetyl* : *see* *m*-Aminoacetanilide.

N : N′-*Diacetyl* : prisms from EtOH.Aq. M.p. 191°.

N-*Benzoyl* : cryst. M.p. 125°.

N : N′-*Dibenzoyl* : needles from AcOH. M.p. 240°. Mod. sol. AcOH. Spar. sol. EtOH.

N-m-*Nitrobenzoyl* : m.p. 142°.

N-p-*Nitrobenzoyl* : light brown needles. M.p. 212°.

N-*Picryl* : orange-red cryst. from Me_2CO. M.p. 206–7°. Sol. Me_2CO. Spar. sol. EtOH, AcOH.

Hazlet, Dornfeld, *J. Am. Chem. Soc.*, 1944, **66**, 1781.

Micewicz, *Chem. Abstracts*, 1928, **22**, 4111.

Briner, Ferrero, Paillard, *Helv. Chim. Acta*, 1926, **9**, 956.

***p*-Phenylenediamine** (1 : 4-*Diaminobenzene*).

Plates from Et_2O. M.p. 140°. B.p. 267°. Sol. EtOH, Et_2O. Sol. 100 parts cold H_2O. Heat of comb. C_v 843·3 Cal.

N-*Me* : *see* *N*-Methyl-*p*-phenylenediamine.

N-*Di-Me* : *see* *p*-Aminodimethylaniline.

N : N′-*Di-Me* : *see* *sym.*-Dimethyl-*p*-phenylenediamine.

N-*Et* : *see* *N*-Ethyl-*p*-phenylenediamine.

N-*Di-Et* : *see* *p*-Aminodiethylaniline.

N-*Phenyl* : *see* *p*-Aminodiphenylamine.

N-*Diphenyl* : *see* *p*-Aminotriphenylamine.

N : N′-*Diphenyl* : *see* *sym.*-Diphenyl-*p*-phenylenediamine.

N : N′-*Tri-Me* : $C_9H_{14}N_2$. MW, 150. Oil. B.p. 265°. Spar. sol. H_2O. *Acetyl* : m.p. 95°.

N : N′-*Tetra-Me* : $C_{10}H_{16}N_2$. MW, 164. Leaflets from EtOH.Aq. or ligroin. M.p. 51°. B.p. 260°. Very sol. EtOH, Et_2O, $CHCl_3$. Sol. ligroin. Spar. sol. cold H_2O. *Methiodide* : leaflets. M.p. 265°.

N : N′-*Tetra-Et* : $C_{14}H_{24}N_2$. MW, 220. Plates from EtOH.Aq. M.p. 52°. B.p. 280°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, ligroin.

N-*Propyl* : $C_9H_{14}N_2$. MW, 150. Leaflets. B.p. 281°. Sol. EtOH, Et_2O, C_6H_6.

N : N′-*Dipropyl* : $C_{12}H_{20}N_2$. MW, 192. Pale yellow oil. B.p. 155·5–156·5°/6 mm. Sol. EtOH. Spar. sol. hot H_2O. N-*Chloroacetyl* : prisms from EtOH. M.p. 121–121·5°.

N-*Butyl* : plates from pet. ether. M.p. 31·5°. B.p. 302·5–303·5°/760 mm. Sol. most org. solvents. Spar. sol. pet. ether. Insol. H_2O. Turns red in air. *B,2HCl* : plates from EtOH. Does not melt below 200°.

N-*Isobutyl* : leaflets from C_6H_6–ligroin. M.p. 39°.

N-*Isoamyl* : cryst. M.p. 31–2°.

N : N′-*Triphenyl* : cryst. from MeOH. M.p. 77–81°. B.p. 205–15°/0·01 mm. Very sol. Me_2CO, AcOEt, CS_2, C_6H_6. N-*Acetyl* : leaflets from EtOH. M.p. 184°.

N : N′-*Tetraphenyl* : leaflets from Me_2CO. M.p. 199–200°. Very sol. C_6H_6. Spar. sol. Et_2O, EtOH, pet. ether. Sol. 90 parts boiling Me_2CO.

N-o-*Tolyl* : *see* 4′-Amino-2-methyldiphenylamine.

N-p-*Tolyl* : *see* 4′-Amino-4-methyldiphenylamine.

N : N′-*Ditolyl* : *see* Ditolyl-*p*-phenylenediamine.

N-*Benzyl* : plates from Et_2O. M.p. 37°. N′-

Acetyl: needles from EtOH. M.p. 141–2°. N : N′-*Diacetyl*: prisms from EtOH.Aq. M.p. 116–17°. N′-*Benzoyl*: plates from EtOH. M.p. 182–3°. N : N′-*Dibenzoyl*: needles. M.p. 124°.

N : N′-*Dibenzyl*: needles from EtOH. M.p. 89–90°. Sol. Et_2O. Spar. sol. EtOH–$FeCl_3$ ⟶ intense red col.

N : N′-*Tetrabenzyl*: needles from AcOH. M.p. 152° (149°). Sol. C_6H_6, $CHCl_3$, CS_2. Spar. sol. EtOH.

N-1-*Naphthyl*: plates. M.p. 80·5–81°. B.p. 275–80°/12 mm. Sol. hot EtOH, C_6H_6. Spar. sol. H_2O. N′-*Acetyl*: needles from EtOH. M.p. 162·5°.

N-2-*Naphthyl*: needles from ligroin. M.p. 94°. Very sol. C_6H_6, EtOH, Et_2O, Me_2CO with blue fluor. *B,2HCl*: m.p. 240°. decomp. N′-*Acetyl*: needles from C_6H_6. M.p. 160°.

Dinaphthyl: *see* Dinaphthyl-*p*-phenylenediamine.

N-*Picryl*: black prisms from AcOEt. M.p. 186–7°. Sol. AcOH, amyl alcohol.

Dibenzylidene: plates from EtOH. M.p. 140°.

N-*Formyl*: brownish needles from H_2O. M.p. 125–7°.

N : N′-*Diformyl*: m.p. 205–7°.

N-*Acetyl*: *see p*-Aminoacetanilide.

N : N′-*Diacetyl*: leaflets from hot AcOH. M.p. 303°.

N-*Benzoyl*: leaflets. M.p. 128°. Sol. EtOH, $CHCl_3$. Spar. sol. H_2O.

N : N′-*Dibenzoyl*: leaflets. Does not melt below 300°. Spar. sol. EtOH, Et_2O, AcOH.

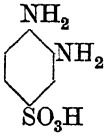
N-m-*Nitrobenzoyl*: golden cryst. from EtOH. M.p. 217–18°.

N-p-*Nitrobenzoyl*: cryst. M.p. 228°.

N-*Carbethoxyl*: *see p*-Aminophenylurethane.

Quick, *J. Am. Chem. Soc.*, 1920, **42**, 1033.
Hazlet, Dornfeld, *J. Am. Chem. Soc.*, 1944, **66**, 1781.

o-Phenylenediamine-4-sulphonic Acid (3 : 4-*Diaminobenzenesulphonic acid*)

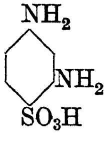


$C_6H_8O_3N_2S$ MW, 188

Needles. Decomp. on melting. Mod. sol. hot H_2O. Turns blue or green in air. $FeCl_3$ ⟶ reddish-brown col.

Nietzki, Lerch, *Ber.*, 1888, **21**, 3221.
Post, Hartung, *Ann.*, 1880, **205**, 100.

m-Phenylenediamine-4-sulphonic Acid (2 : 4-*Diaminobenzenesulphonic acid*)

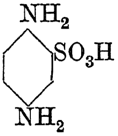


$C_6H_8O_3N_2S$ MW, 188

Plates and prisms from H_2O. Spar. sol. cold H_2O. Slowly turns brown in air.

Post, Hartung, *Ann.*, 1880, **205**, 107.
Hunter, Sprung, *J. Am. Chem. Soc.*, 1931, **53**, 1440.

p-Phenylenediamine-2-sulphonic Acid (2 : 5-*Diaminobenzenesulphonic acid*)



$C_6H_8O_3N_2S$ MW, 188

Plates + $2H_2O$ from H_2O, needles from H_2O with trace H_2SO_4. Mod. sol. H_2O. Insol. EtOH, Et_2O, C_6H_6.

N-*Di-Me*: sol. H_2O. $FeCl_3$ ⟶ red col.

Anilide: leaflets from H_2O. M.p. 171°. *B,HCl*: needles. M.p. 215° decomp.

A.G.F.A., D.R.P., 204,972, (*Chem. Zentr.*, 1909, I, 475).
Kalle, D.R.P., 124,907, (*Chem. Zentr.*, 1901, II, 1103).
Fischer, *Ber.*, 1891, **24**, 3789.
Eger, *Ber.*, 1889, **22**, 848.

Phenylene Dimercaptan.

See Dithiocatechol, Dithiohydroquinone *and* Dithioresorcinol.

Phenylenedimethyldiamine.

See sym.-Dimethylphenylenediamine.

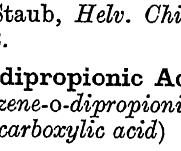
Phenylenedinaphthyldiamine.

See Dinaphthylphenylenediamine.

Phenylenediphenyldiamine.

See sym.-Diphenylphenylenediamine.

m-Phenylenedipropiolic Acid (m-*Benzenedipropiolic acid*)



$C_{12}H_6O_4$ MW, 214

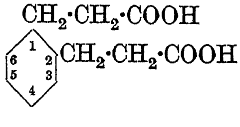
M.p. 209°.

Di-Me ester: m.p. 93°.

Di-Et ester: m.p. 56°.

Ruggli, Staub, *Helv. Chim. Acta.*, 1936, **19**, 962.

o-Phenylenedipropionic Acid (o-*Xylylenediacetic acid, benzene*-o-*dipropionic acid*, o-*diethylbenzene*-ω : ω′-*dicarboxylic acid*)



$C_{12}H_{14}O_4$ MW, 222

Needles from H_2O, prisms from EtOH.Aq. M.p. 171° (168°, 162°).

Di-Et ester: $C_{16}H_{22}O_4$. MW, 278. B.p. 200–2°/12 mm.

Diamide: $C_{12}H_{16}O_2N_2$. MW, 220. Needles from EtOH. M.p. 195°.

Titley, *J. Chem. Soc.* 1928, 2578.
Fries, Bestian, *Ber.*, 1936, **69**, 720.

***m*-Phenylenedipropionic Acid** (m-*Xylylenediacetic acid, benzene*-m-*dipropionic acid*, m-*diethylbenzene*-ω : ω'-*dicarboxylic acid*).

Plates from H_2O. M.p. 146–7°. Mod. sol. EtOH, Et_2O.

Di-Me ester: $C_{14}H_{18}O_4$. MW, 250. Leaflets from MeOH.Aq. M.p. 51°. Very sol. EtOH, Et_2O, C_6H_6.

Di-Et ester: liq. B.p. 247–50°/60 mm.

Kipping, *J. Chem. Soc.*, 1888, **53**, 33.

***p*-Phenylenedipropionic Acid** (p-*Xylylenediacetic acid, benzene*-p-*dipropionic acid*, p-*diethylbenzene*-ω : ω'-*dicarboxylic acid*).

Cryst. from MeOH. M.p. 223–4°. Spar. sol. EtOH. Insol. H_2O.

Di-Me ester: plates from MeOH. M.p. 115°. Spar. sol. cold MeOH.

Kipping, *J. Chem. Soc.*, 1888, **53**, 39.
Pohl, *J. prakt. Chem.*, 1934, **141**, 44.

Phenyleneditolyldiamine.
See Ditolylphenylenediamine.

***o*-Phenyleneurea.**
See Benziminazolone.

Phenylerythrene.
See Phenyl-1 : 3-butadiene.

Phenylethane.
See Ethylbenzene.

Phenylethanolamine.
See Hydroxyaminoethylbenzene.

Phenyl *p*-ethoxyphenacyl Ether.
See under *p*-Hydroxyphenacyl Alcohol.

Phenyl ethoxystyryl Ketone.
See under Hydroxychalkone.

β-Phenylethylacetylene.
See 4-Phenyl-1-butyne.

1-Phenylethyl Alcohol.
See Methylphenylcarbinol.

2-Phenylethyl Alcohol (*Benzylcarbinol*, β-*hydroxyethylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2OH$

$C_8H_{10}O$ MW, 122

Found in fresh rose petals and neroli oil. B.p. 219–21°/750 mm., 104°/14 mm., 98–100°/12 mm. D_4^{25} 1·0235. $n_D^{16\cdot8}$ 1·5337. Mod. sol. H_2O, EtOH.Aq. Used in perfumery, as also are its esters.

Me ether: *see* Methyl phenylethyl Ether.

Phenyl ether: phenyl phenylethyl ether. $C_{14}H_{14}O$. MW, 198. B.p. 166°/14 mm.

Formyl: b.p. 94°/9 mm. D^{15} 1·054.

Acetyl: b.p. 224°, 118–20°/13 mm. D^{15} 1·038. n_D 1·5108.

Methoxyacetyl: b.p. 149°/18 mm. D_4^{17} 1·0806. n_D^{17} 1·5000.

Oxalate: cryst. from 80% EtOH. M.p. 51·5°.

p-*Nitrobenzoyl*: m.p. 62–3°.

Allophanate: m.p. 186°.

Phenylurethane: m.p. 79–80°.

Skita, Ritter, *Ber.*, 1911, **43**, 3398.
Bouveault, Blanc, *Bull. soc. chim.*, 1904, **31**, 674.
Skita, *Ber.*, 1915, **48**, 1694.
Skita, Mayer, *Ber.*, 1912, **45**, 3584.
Leonard, *J. Am. Chem. Soc.*, 1925, **47**, 1774.

Phenylethylamine.
See Aminoethylbenzene.

β-Phenylethylbenzylamine.
See Benzyl-phenylethylamine.

Phenylethyl bromide.
See Bromoethylbenzene.

Phenylethyl chloride.
See Chloroethylbenzene.

Phenylethylene.
See Styrene.

Phenylethylene bromohydrin.
See Styrene bromohydrin.

Phenylethylene chlorohydrin.
See Styrene chlorohydrin.

Phenylethylenediamine (αβ-*Diaminoethylbenzene*)

$C_6H_5{\cdot}CH(NH_2){\cdot}CH_2NH_2$

$C_8H_{12}N_2$ MW, 136

l-.

M.p. 4–5°. B.p. 104°/1–2 mm. D^{18} 1·034. $[\alpha]_D - 35{\cdot}2°$.

N : N'-*Diacetyl*: cryst. from AcOEt. M.p. 174°.

N : N'-*Dibenzoyl*: cryst. from EtOH. M.p. 227°.

d-*Tartrate*: m.p. 220° decomp. $[\alpha]_D + 40{\cdot}8°$ in H_2O.

dl-.

B.p. 243–6°, 156–7°/42 mm. Very sol. H_2O. Sol. most org. solvents.

Diacetyl: cryst. from AcOEt. M.p. 159° (152°).

Triacetyl: plates from H_2O. M.p. 166°.

Dibenzoyl: leaflets from EtOH. M.p. 217°.

Picrate: yellow cryst. M.p. 160°. Insol. C_6H_6, ligroin.

B,2HCl: needles. Does not melt below 260°.

Thurston, Shriner, *J. Org. Chem.*, 1937, **2**, 192.
Reihlen, Hessling, Hühn, Weinbrenner, *Ann.*, 1932, **493**, 25.
Kanewskaja, *J. prakt. Chem.*, 1932, **132**, 340.
Reihlen, Weinbrenner, Hessling, *Ann.*, 1932, **494**, 157.
Feist, Arnstein, *Ber.*, 1893, **28**, 425, 3172.

Phenylethylene dibromide.
See Styrene dibromide.

Phenylethylene dichloride.
See Styrene dichloride.

Phenylethylene Glycol.
See Styrene Glycol.

Phenylethylene oxide.
See Styrene oxide.

Phenylethylhydrazine (*Phenethylhydrazine, ω-hydrazinoethylbenzene*)

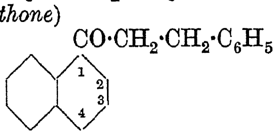
$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}NH_2$

$C_8H_{12}N_2$ MW, 136

B.p. 137–9°/12–13 mm.
B,HCl: plates from EtOH. M.p. 171°.
Dibenzoyl: needles from 50% EtOH. M.p. 144–5°.

Votoček, Lemings, *Chem. Zentr.*, 1932, II, 1613.

Phenylethyl 1-naphthyl Ketone (*ω-Benzyl-α-acetonaphthone*)



$C_{19}H_{16}O$ MW, 260

Leaflets from 70% EtOH. M.p. 93° (53–4°). B.p. 240–60°/25 mm. Sol. conc. H_2SO_4 with yellow col. turning brownish-red on warming.
Oxime: cryst. from EtOH. M.p. 120°.

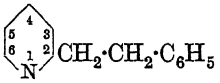
Albrecht, *Monatsh.*, 1914, **35**, 1498.
Bergmann, Weiss, *Ber.*, 1931, **64**, 1492.

Phenylethyl 2-naphthyl Ketone (*ω-Benzyl-β-acetonaphthone*).
M.p. 63°. B.p. 217–19°.

Mastagli, *Ann. chim.*, 1938, **10**, 369.

Phenylethylpiperidine.
See Stilbazoline.

2-Phenylethylpyridine (*Dihydro-α-stilbazole, phenyl-2-pyridylethane*)



$C_{13}H_{13}N$ MW, 183

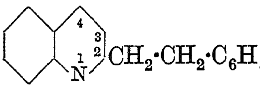
Liq. with sweet odour. F.p. — 3°. B.p. 289·5°/766 mm. D_4^0 1·0465. Very sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Volatile in steam.
B,HCl: hygroscopic needles. M.p. 102–4°.
B,HAuCl₄: yellow needles from dil. HCl. M.p. 149–50°.
B,HCl,HgCl₂: cryst. M.p. 149°.
B₂,H₂PtCl₆: reddish-yellow needles. M.p. 185–6°.
Picrate: m.p. 125·5–127°.

Baurath, *Ber.*, 1888, **21**, 821.
Bergstrom, Norton, Seibert, *J. Org. Chem.*, 1945, **10**, 454.

4-Phenylethylpyridine (*γ-Dihydrostilbazole, phenyl-4-pyridylethane*).
Needles from EtOH. M.p. 69–71° (65°).
B,HCl: cryst. from EtOH–Et_2O. M.p. 180°.
B,HI: brownish-red leaflets from H_2O. Decomp. about 150°.
B,HAuCl₄: yellow leaflets. M.p. 183–5° (166°).
B₂,H₂PtCl₆: brown leaflets. M.p. 214°.
Picrate: yellow needles. M.p. 162–3°.

Friedländer, *Ber.*, 1905, **38**, 2837.
Fels, *Ber.*, 1904, **37**, 2147.
Bergstrom, Norton, Seibert, *J. Org. Chem.*, 1945, **10**, 453.

2-Phenylethylquinoline (*Phenyl-2-quinolylethane*)



$C_{17}H_{15}N$ MW, 233

Cryst. from C_6H_6 or ligroin. M.p. 28°. B.p. 214–19°/14 mm. Spar. volatile in steam.
Picrate: pale yellow prisms. M.p. about 130°.

Heymann, Koenigs, *Ber.*, 1888, **21**, 1426.
Ziegler, Zeiser, *Ann.*, 1931, **485**, 190.
Bergstrom, Norton, Seibert, *J. Org. Chem.*, 1945, **10**, 455.

4-Phenylethylquinoline (*Phenyl-4-quinolylethane*).
Cryst. from EtOH.Aq. or ligroin. M.p. 100–1°.
Picrate: m.p. 183–6°.

Heymann, Koenigs, *Ber.*, 1888, **21**, 1427.
Bergstrom, Norton, Seibert, *J. Org. Chem.*, 1945, **10**, 456.

α-Phenylethyl-succinic Acid (*3-Phenylbutane-1 : 2-dicarboxylic acid*)

$$C_6H_5{\cdot}\underset{}{\overset{CH_3}{CH}}{\cdot}\underset{CH_2{\cdot}COOH}{CH}{\cdot}COOH$$

$C_{12}H_{14}O_4$ MW, 222

Needles from C_6H_6 or hot H_2O. M.p. 144–146·5°. Sol. EtOH, Et_2O. Spar. sol. cold C_6H_6.

Stobbe, *Ann.*, 1899, **308**, 127.

β-Phenylethyl-succinic Acid (*4-Phenylbutane-1 : 2-dicarboxylic acid*)

$$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}\underset{CH_2{\cdot}COOH}{CH}{\cdot}COOH$$

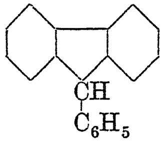
$C_{12}H_{14}O_4$ MW, 222

Cryst. from H_2O or C_6H_6. M.p. 136°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6. Insol. CS_2, low-boiling pet. ether.
Mono-NH₄ salt: leaflets from H_2O. M.p. 185° decomp.
Anhydride: $C_{12}H_{12}O_3$. MW, 204. Cryst. from pet. ether. M.p. 56°. Sol. $CHCl_3$, C_6H_6. Spar. sol. CS_2, pet. ether.

Thiele, Meisenheimer, *Ann.*, 1899, **306**, 257, 261.

Phenylethynyl bromide.
See ω-Bromophenylacetylene.

9-Phenylfluorene (*Phenyldiphenylene-methane*)



$C_{19}H_{14}$ MW, 242

Needles or leaflets from EtOH or C_6H_6. M.p. 148° (145°). Sol. AcOH, $CHCl_3$, C_6H_6, pet. ether, hot EtOH. Spar. sol. Et_2O. Alc. and benzene solutions show blue fluor. $CrO_3 \longrightarrow$ *o*-benzoylbenzoic acid.

Vorländer, Pritzsche, *Ber.*, 1913, **46**, 1796.
Kliegl, *Ber.*, 1905, **38**, 287.
Koelsch, *J. Am. Chem. Soc.*, 1934, **56**, 482.
Seibert, Bergstrom, *J. Org. Chem.*, 1945, **10**, 549.

Phenyl formate

$H{\cdot}CO{\cdot}OC_6H_5$

$C_7H_6O_2$ MW, 122

B.p. 173° slight decomp., 107°/25 mm. D^0 1·0879.

Seifert, *J. prakt. Chem.*, 1885, **31**, 467.
Auger, *Compt. rend.*, 1904, **139**, 799.
Adickes, Brunnert, Lücker, *J. prakt. Chem.*, 1931, **130**, 174.

Phenylformylacetanilide.

See under Phenylformylacetic Acid.

Phenylformylacetic Acid (*Phenylaldehydo-acetic acid, β-hydroxyatropic acid, α-hydroxy-methylenephenylacetic acid, 2-hydroxy-1-phenyl-acrylic acid, phenylmalonaldehydic acid*)

$$\underset{\text{aldo-form}}{C_6H_5{\cdot}\overset{\displaystyle CHO}{\overset{|}{C}H}{\cdot}COOH} \rightleftharpoons \underset{\text{enol-form}}{C_6H_5{\cdot}\overset{\displaystyle CH{\cdot}OH}{\overset{\|}{C}}{\cdot}COOH}$$

$C_9H_8O_3$ MW, 164

The acid is unknown in the free state.

Me ester: $C_{10}H_{10}O_3$. MW, 178. α(*enol*)-form: leaflets. M.p. 40–1°. β(probably *aldo*)-form: leaflets from C_6H_6. M.p. 91–3° (87°). *Semicarbazone*: m.p. 159–60°. *Aldo-enol* mixture: b.p. 153–5°/18 mm. n^{20} 1·52425.

Et ester: $C_{11}H_{12}O_3$. MW, 192. α(mainly *enol*)-form: b.p. 135°/15 mm. Alc. sol. $\longrightarrow$ bluish-violet col. with $FeCl_3$. *Formyl*: m.p. 87°. *Acetyl*: b.p. 184°/18 mm. β(probably *aldo*)-form: m.p. 50° (70°). *Semicarbazone*: m.p. 162–5° (130–1°). *Aldo-enol* mixture: b.p. 150–1°/18 mm. n^{20} 1·532. *γ-form*: plates. M.p. 108–10°.

Nitrile: α-formylbenzyl cyanide, α-cyano-phenylacetaldehyde. C_9H_7ON. MW, 145. Leaflets from EtOH. M.p. 165–6° (157–8°). Sol. EtOH, C_6H_6, hot H_2O.

Anilide: phenylformylacetanilide. Exists in two modifications. (α-) Plates from pet. ether. M.p. 68°. (β-) Triangular plates from Et_2O or AcOH. M.p. 98°.

Piperidide: exists in two modifications. (α-) M.p. 104–6°. (β-) M.p. 122°.

Décombe, *Ann. chim.*, 1932, **18**, 90, 113.
Wislicenus, Erbe, *Ann.*, 1920, **421**, 119.
Tiffeneau, Levy, *Chem. Abstracts*, 1930, **24**, 2450.
Ghosh, *J. Chem. Soc.*, 1916, **109**, 113.
Wislicenus, *Ann.*, 1917, **413**, 206.

Phenylfumaric Acid

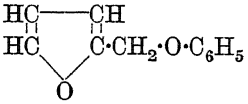
$$\begin{array}{c} C_6H_5{\cdot}C{\cdot}COOH \\ \| \\ HOOC{\cdot}CH \end{array}$$

$C_{10}H_8O_4$ MW, 192

Needles from C_6H_6. M.p. 128–9°. Very sol. H_2O. Spar. sol. C_6H_6. Above m.p. $\longrightarrow$ phenyl-maleic anhydride. Reduces $KMnO_4$.Aq. slowly.

Almström, *Ber.*, 1915, **48**, 2009; *Ann.*, 1916, **411**, 375.

Phenyl furfuryl Ether



$C_{11}H_{10}O_2$ MW, 174

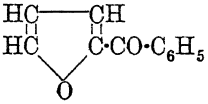
B.p. 139–40°/20 mm. D_{15}^{17} 1·124. n_D^{17} 1·5535.

Normant, *Ann. chim.*, 1942, **17**, 335.

Phenyl furyl Diketone.

See Benzfuril.

Phenyl α-furyl Ketone (*2-Benzoylfuran, α-benzofuran*)



$C_{11}H_8O_2$ MW, 172

B.p. 285°, 186°/46 mm., 164°/19 mm. D^{20} 1·1732. n_D^{20} 1·6055 (1·5798).

Oxime: yellow needles from C_6H_6–pet. ether. M.p. 138° (132°). Very sol. org. solvents.

Semicarbazone: m.p. 182°.

Asahina, Murayama, *Arch. Pharm.*, 1914, **252**, 448.
Marquis, *Bull. soc. chim.*, 1900, **23**, 33.
Gilman, Hewlett, *Chem. Abstracts*, 1930, **24**, 1640.
Hartough, Kosak, *J. Am. Chem. Soc.*, 1947, **69**, 3095, 3098.

Phenyl furylvinyl Ketone.

See Furfurylideneacetophenone.

Phenyl-gamma Acid.

See *N*-Phenyl-2-amino-8-naphthol-6-sulphonic Acid.

2-Phenylglutaconic Acid (*2-Phenylpropyl-ene-1 : 3-dicarboxylic acid*)

$$HOOC{\cdot}CH_2{\cdot}\overset{\displaystyle C_6H_5}{\overset{|}{C}}{:}CH{\cdot}COOH$$

$C_{11}H_{10}O_4$ MW, 206

Prismatic plates from AcOEt. M.p. 154–5°. Above m.p. ⟶ anhydride. P + HI at 150° ⟶ 2-phenylglutaric acid.

Mono-Et ester: $C_{13}H_{14}O_4$. MW, 234. Needles from C_6H_6–pet. ether. M.p. 78°. Dist. ⟶ anhydride.

Di-Et ester: $C_{15}H_{18}O_4$. MW, 262. B.p. 186–7°/11 mm., 167–8°/5 mm. D_4^{20} 1·1014. n_D^{20} 1·5240.

Monoamide: $C_{11}H_{11}O_3N$. MW, 205. Needles from AcOEt. M.p. 138°.

Mononitrile: $C_{11}H_9O_2N$. MW, 187. Leaflets from EtOH or AcOH. M.p. 256–7°. Spar. sol. H_2O, EtOH.

Anhydride: $C_{11}H_8O_3$. MW, 188. Leaflets from AcOEt. M.p. 206°.

Imide: $C_{11}H_9O_2N$. MW, 187. Prisms from AcOH, leaflets from EtOH. M.p. 256–7°. Spar. sol. boiling EtOH. Very spar. sol. H_2O, Et_2O. Alc. $FeCl_3$ ⟶ red col.

Monoanilide: cryst. from AcOEt. M.p. 174°.

Mono-p-*toluidide*: cryst. from AcOEt. M.p. 184°.

Michael, *J. prakt. Chem.*, 1894, **49**, 22.
Ruhemann, *J. Chem. Soc.*, 1899, **75**, 252.
Feist, Pomme, *Ann.*, 1909, **370**, 74.
Guareschi, *Chem. Zentr.*, 1907, I, 459; 1901, I, 821.
Bland, Thorpe, *J. Chem. Soc.*, 1912, **101**, 868.
Gidvani, Kon, *J. Chem. Soc.*, 1932, 2448.

3-Phenylglutaconic Acid (3-*Phenylpropylene*-1 : 3-*dicarboxylic acid*)

C_6H_5
HOOC·CH·CH:CH·COOH

$C_{11}H_{10}O_4$ MW, 206

Cryst. from EtOH. M.p. 164°.

Di-Et ester: b.p. 220°/80 mm. D^{30} 1·0712. n_D^{30} 1·50923.

Phalnikar, Nargund, *Chem. Zentr.*, 1936, I, 4556.

2-Phenylglutamic Acid

C_6H_5
$HOOC·CH_2·CH·CH(NH_2)·COOH$

$C_{11}H_{13}O_4N$ MW, 223

Plates from H_2O. M.p. 179°. Insol. EtOH.

N-*Benzoyl*: needles from H_2O. M.p. 171–2°.

Harington, *J. Biol. Chem.*, 1925, **64**, 29.

3-Phenylglutamic Acid

C_6H_5
$HOOC·CH·CH_2·CH(NH_2)·COOH$

$C_{11}H_{13}O_4N$ MW, 223

Rhombic cryst. M.p. 185° decomp. Spar. sol. H_2O.

v. Beznák, *Biochem. Z.*, 1929, **205**, 414.

1-Phenylglutaric Acid (1-*Phenylpropane*-1 : 3-*dicarboxylic acid*)

C_6H_5
$HOOC·CH_2·CH_2·CH·COOH$

$C_{11}H_{12}O_4$ MW, 208

Cryst. from C_6H_6 or Et_2O–pet. ether. M.p. 82–3°. Dist. in vacuo ⟶ anhydride.

Anhydride: $C_{11}H_{10}O_3$. MW, 190. Needles from Et_2O. M.p. 95°. B.p. 218–30°/13 mm.

Fichter, Merckens, *Ber.*, 1901, **34**, 4175.

2-Phenylglutaric Acid (*Benzylidenediacetic acid*, 2-*phenylpropane*-1 : 3-*dicarboxylic acid*)

C_6H_5
$HOOC·CH_2·CH·CH_2·COOH$

$C_{11}H_{12}O_4$ MW, 208

Plates from H_2O, prisms from C_6H_6. M.p. 140°. Sol. EtOH, Et_2O, AcOEt, $CHCl_3$. Spar. sol. C_6H_6. Insol. pet. ether. $k = 7·7 \times 10^{-5}$ at 25°. Above m.p. ⟶ anhydride.

Di-Me ester: $C_{13}H_{16}O_4$. MW, 236. Prisms or needles from MeOH. M.p. 86–7°.

Di-Et ester: $C_{15}H_{20}O_4$. MW, 264. B.p. 188–9°/13 mm.

Dichloride: $C_{11}H_{10}O_2Cl_2$. MW, 245. M.p. 46°. B.p. 178–80°/18 mm.

Anhydride: $C_{11}H_{10}O_3$. MW, 190. Cryst. from C_6H_6. M.p. 105°. B.p. 217–19°/15 mm. Sol. Et_2O, $CHCl_3$, C_6H_6. Insol. pet. ether.

Imide: $C_{11}H_{11}O_2N$. MW, 189. Leaflets from H_2O, EtOH or C_6H_6. M.p. 173–4°.

Monoanilide: needles from EtOH.Aq. M.p. 171° (168°).

Mono-p-*toluidide*: needles. M.p. 154–5°.

Anil: needles from EtOH. M.p. 223°.

Michael, *J. prakt. Chem.*, 1887, **35**, 352.
Herrmann, Vorländer, *Chem. Zentr.*, 1899, I, 730.
Avery, Bouton, *Am. Chem. J.*, 1898, **20**, 513.
v. Braun, Weissbach, *Ber.*, 1931, **64**, 1787.

1-Phenylglyceric Acid (1 : 2-*Dihydroxy*-1-*phenylpropionic acid*, *atroglyceric acid*)

C_6H_5
$HO·CH_2·C(OH)·COOH$

$C_9H_{10}O_4$ MW, 182

Cryst. from H_2O. M.p. 146°. Mod. sol. hot H_2O. Heat ⟶ phenylacetaldehyde.

Nitrile: $C_9H_9O_2N$. MW, 163. Needles from Et_2O. M.p. 55–7° decomp.

Plöchl, Blümlein, *Ber.*, 1883, **16**, 1292.
Fittig, Kast, *Ann.*, 1881, **206**, 30.
McKenzie, Wood, *J. Chem. Soc.*, 1919, **115**, 838.
Craig, Henze, *J. Org. Chem.*, 1945, **10**, 19.

2-Phenylglyceric Acid (αβ-*Dihydroxyhydrocinnamic acid*)

$C_6H_5·CH(OH)·CH(OH)·COOH$

$C_9H_{10}O_4$ MW, 182

Exists in two stereoisomeric forms.

(i) *d*-.

Needles or plates from C_6H_6. M.p. 95°. Sol. 10 parts Et_2O at 20°. $[\alpha]_D^{20}$ + 26·1° in H_2O, + 21·15° in EtOH, + 27·5° in Me_2CO.

l-.

Cryst. M.p. 97–8°. $[\alpha]_D$ — 25·6°.

dl-.

Needles from Et_2O. M.p. 122°. Sol. 15 parts Et_2O. $k = 2·35 \times 10^{-4}$.

Me ester: $C_{10}H_{12}O_4$. MW, 196. Cryst. M.p. 87°. B.p. 110°/0·1 mm. *Dibenzoyl*: cryst. from EtOH. M.p. 113·5°.

Monoacetyl: cryst. from H_2O. M.p. 158°.

Dibenzoyl: needles from C_6H_6. M.p. 187° decomp. Sol. EtOH. Spar. sol. C_6H_6, hot H_2O.

Et ester: prisms from toluene. M.p. 109°.

Phenylhydrazide: m.p. 177°.

(ii) *d*-.

Plates from H_2O. M.p. 166–7° (164°). Sol. 200 parts Et_2O, 25 parts H_2O at 20°. $[\alpha]_D^{20}$ + 39·6° in H_2O.

l-.

Plates from H_2O. M.p. 166–7° (164°). Sublimes in high vacuum. $[\alpha]_D^{20}$ — 39·6° in H_2O, — 30·5° in EtOH, — 36·4° in Me_2CO.

dl-.

Plates from H_2O, leaflets from Et_2O or C_6H_6. M.p. 141–2°. Very sol. H_2O, EtOH. Mod. sol. Me_2CO. Spar. sol. $CHCl_3$, CS_2. Insol. C_6H_6, ligroin. Sol. 75 parts Et_2O at 20°. $k = 2·54 \times 10^{-4}$. At 160° ⟶ phenylacetaldehyde.

Me ester: cryst. M.p. 67°. B.p. 114°/0·1 mm.

Et ester: *dibenzoyl*, needles from EtOH. M.p. 85°.

Monoacetyl: cryst. from H_2O. M.p. 93·5°.

Diacetyl: leaflets + $\frac{1}{2}H_2O$ from H_2O. M.p. 88–9°.

Amide: $C_9H_{11}O_3N$. MW, 181. Leaflets from EtOH. M.p. 161–2°. Sol. EtOH, Me_2CO, hot H_2O.

Phenylhydrazide: m.p. 215°.

Anschütz, Kinnicutt, *Ber.*, 1879, **12**, 539.
Plöchl, Mayer, *Ber.*, 1897, **30**, 1601.
Lipp, *Ber.*, 1883, **16**, 1289.
Riiber, *Ber.*, 1908, **41**, 2413.
Riiber, Berner, *Ber.*, 1917, **50**, 894.
Berner, *Chem. Zentr.*, 1919, III, 777.
English, Gregory, *J. Am. Chem. Soc.*, 1947, **69**, 2122.

1-Phenylglycerol (*Stycerine*, 1 : 2 : 3-*trihydroxy*-1-*phenylpropane*)

HO·CH_2·CH(OH)·CH(OH)·C_6H_5

$C_9H_{12}O_3$ MW, 168

Cryst. from EtOH. M.p. 100·5° (98–9°). B.p. 184–6°. $D_4^{13·5}$ 1·2213. n_D^{15} 1·5600.

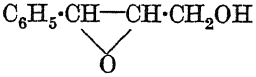
1-*Me ether*: $C_{10}H_{14}O_3$. MW, 182. M.p. 68°.

2-*Me ether*: m.p. 44°. B.p. 153–6°/5–6 mm.

3-*Me ether*: needles. M.p. 54°. B.p. 159°/8 mm. Sol. H_2O, EtOH, Et_2O, C_6H_6. Insol. pet. ether.

Platt, Hibbert, *Chem. Zentr.*, 1933, I, 3923.
Tiffeneau, Neuberg-Rabinovitch, Cahmann, *Bull. soc. chim.*, 1935, **2**, 1869.
See also Prévost, Losson, *Compt. rend.*, 1934, **198**, 659.

Phenylglycide (*Phenylglycidol*)



$C_9H_{10}O_2$ MW, 150

Waxy cryst. M.p. 26·5°. B.p. 134–5°/4–5 mm. n_D^{27} 1·5427.

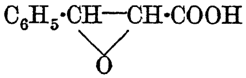
Me ether: $C_{10}H_{12}O_2$. MW, 164. B.p. 130–7°/21–3 mm. n_D^{20} 1·5170.

Acetyl: b.p. 129–32°/3 mm. n_D^{22} 1·5208.

Phenylurethane: cryst. from C_6H_6–pet. ether. M.p. 87°.

Jahn, Hibbert, *Chem. Zentr.*, 1933, II, 1018.
Darmon, Weill, *Bull. soc. chim.*, 1941, **8**, 407.

Phenylglycidic Acid



$C_9H_8O_3$ MW, 164

l-.

Na salt: $[\alpha]_D$ — 157·9° in H_2O.

dl-.

Prisms. M.p. 83–4°. B.p. 128–30°/4–5 mm. Spar. sol. H_2O. Stable at ord. temps. Above m.p. ⟶ phenylacetaldehyde. Dil. H_2SO_4 ⟶ phenylacetaldehyde + 2-phenylglyceric acid (both forms).

Et ester: $C_{11}H_{12}O_3$. MW, 192. B.p. 279·5° slight decomp.

Anilide: needles from EtOH. M.p. 142°.

Erlenmeyer, *Ber.*, 1906, **39**, 789.
Dieckmann, *Ber.*, 1910, **43**, 1035.
Erdmann, D.R.P., 107,228.
I.G., F.P., 715,657, (*Chem. Zentr.*, 1932, II, 2747).
Kaufmann, D.R.P., 515,034, (*Chem. Zentr.*, 1931, I, 1829).
Darzens, Levy, *Compt. rend.*, 1937, **204**, 272.

***C*-Phenylglycine.**

See α-Aminophenylacetic Acid.

Phenylglycine (*Anilinoacetic acid*)

C_6H_5·NH·CH_2·COOH

$C_8H_9O_2N$ MW, 151

Cryst. M.p. 127–8°. Mod. sol. H_2O. Less sol. Et_2O. $k = 3{\cdot}8 \times 10^{-6}$ at 25°. Na and K salts sol. H_2O, Ca salt spar. sol. H_2O, Fe and Cu salts insol. H_2O. Intermediate in manufacture of indigo.

Me ester: $C_9H_{11}O_2N$. MW, 165. Needles. M.p. 48°. Sol. EtOH, Et_2O. Insol. H_2O. Volatile in steam.

Et ester: $C_{10}H_{13}O_2N$. MW, 179. Leaflets. M.p. 58°. B.p. 273–4°, 163°/18 mm., 140°/8 mm. Sol. EtOH, Et_2O. Spar. sol. hot H_2O. Volatile in steam. N-*Carboethoxyl*: thick liq. B.p. 177–8°/14 mm.

Isoamyl ester: $C_{13}H_{19}O_2N$. MW, 221. Leaflets from C_6H_6. M.p. 37–9°. Sol. Et_2O, C_6H_6, amyl alcohol. Insol. H_2O. N-*Isoamyl*: $C_{18}H_{29}O_2N$. MW, 291. B.p. 215–16°/20 mm.

Phenyl ester: $C_{14}H_{13}O_2N$. MW, 227. Leaflets from cold EtOH.Aq. M.p. 82–3°. Sol. cold EtOH, Et_2O, $CHCl_3$, C_6H_6. Hot EtOH $\longrightarrow$ phenylglycine ethyl ester.

p-*Tolyl ester*: $C_{15}H_{15}O_2N$. MW, 241. Pale yellow needles from pet. ether. M.p. 109°.

Amide: $C_8H_{10}ON_2$. MW, 150. Needles from H_2O, leaflets from EtOH. M.p. 136°. Sol. EtOH, Me_2CO, hot H_2O. Spar. sol. Et_2O.

Methylamide: m.p. 118°.

Ethylamide: m.p. 53–4°.

Anilide: needles from EtOH. M.p. 113°. Sol. EtOH, Et_2O, hot H_2O.

p-*Toluidide*: needles. M.p. 171–2° (165°). Sol. EtOH, Et_2O.

Nitrile: $C_8H_8N_2$. MW, 132. Plates from ligroin–Et_2O. M.p. 48°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O, ligroin.

Hydrazide: leaflets from EtOH. M.p. 126·5°. Sol. warm H_2O, EtOH. Spar. sol. Et_2O.

Piperidide: m.p. 102–3°.

N-*Me*: *see N*-Phenylsarcosine.

N-*Et*: $C_{10}H_{13}O_2N$. MW, 179. Oil. *Et ester*: $C_{12}H_{15}O_2N$. MW, 207. B.p. 280°/759 mm., 178°/42 mm. Turns brown in air. *Isoamyl ester*: $C_{15}H_{23}O_2N$. MW, 249. B.p. 187°/18 mm. *Amide*: $C_{10}H_{14}ON_2$. MW, 178. M.p. 114–15°. *Nitrile*: $C_{10}H_{12}N_2$. MW, 160. Cryst. M.p. about 24°. B.p. 183°/20 mm., 150–1°/13 mm.

N-*Phenyl*: *see* Diphenylglycine.

N-*Formyl*: *see* Formylphenylglycine.

N-*Acetyl*: *N*-phenylaceturic acid. $C_{10}H_{11}O_3N$. MW, 193. Leaflets from H_2O. M.p. 194°. Sol. EtOH, AcOH, AcOEt. Spar. sol. H_2O, Et_2O, $CHCl_3$, C_6H_6, ligroin. $k = 2{\cdot}6 \times 10^{-4}$ at 25°. *Et ester*: m.p. 194–5°.

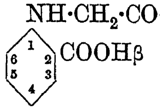
N-*Chloroacetyl*: plates or prisms from C_6H_6. M.p. 132–3°. Sol. EtOH, C_6H_6. $k = 3{\cdot}4 \times 10^{-4}$ at 25°. *Me ester*: prisms from ligroin. M.p. 59–60°.

N-*Bromoacetyl*: leaflets from H_2O. M.p. 153° decomp. $k = 3{\cdot}4 \times 10^{-4}$ at 25°. *Me ester*: leaflets from ligroin. M.p. 71°.

N-*Benzoyl*: *N*-phenylhippuric acid. Amorph. M.p. 63°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

Wohl, Blank, D.R.P., 167,698, (*Chem. Zentr.*, 1906, I, 1069).
Mai, *Ber.*, 1902, **35**, 580.
Curtius, *J. prakt. Chem.*, 1888, **38**, 436.
Lippmann, D.R.P., 163,515, (*Chem. Zentr.*, 1905, II, 1475).
Bischoff, *Ber.*, 1889, **22**, 1809.
Badische, D.R.P., 142,559, (*Chem. Zentr.*, 1903, II, 81).
Paal, Otten, *Ber.*, 1890, **23**, 2592.
Fischer, Gluud, *Ann.*, 1909, **369**, 266.
Thorpe, Wood, *J. Chem. Soc.*, 1913, **103**, 1606.
Stollé, *J. prakt. Chem.*, 1914, **90**, 274.
Wessely, *Z. physiol. Chem.*, 1925, **146**, 72.
B.D.C., B.P. 188,933, (*Chem. Abstracts*, 1923, **17**, 1646).
Rebuffat, *Gazz. chim. ital.*, 1887, **17**, 232.

Phenylglycine-o-carboxylic Acid (*Anthranilinoacetic acid*)



$C_9H_9O_4N$ MW, 195

Needles from MeOH. M.p. 218–20°. Sol. EtOH, Et_2O, AcOH. Mod. sol. hot H_2O. Insol. $CHCl_3$, C_6H_6. Alc. sol. shows blue fluor. Intermediate in manufacture of indigo.

α-*Me ester*: $C_{10}H_{11}O_4N$. MW, 209. M.p. 160°.

β-*Me ester*: m.p. 182°. α-*Amide*: $C_{10}H_{12}O_3N_2$. MW, 208. M.p. 195°. α-*Anilide*: m.p. 140–2°.

Di-Me ester: $C_{11}H_{13}O_4N$. MW, 223. Leaflets. M.p. 97°. Volatile in steam. C_6H_6 and Et_2O sols. show blue fluor. N-*Acetyl*: m.p. 83°. B.p. 205–12°/ 30 mm.

α-*Et ester*: $C_{11}H_{13}O_4N$. MW, 223. Needles from EtOH. M.p. 152°. Sol. $CHCl_3$. β-*Me ester*: $C_{12}H_{15}O_4N$. MW, 237. M.p. 48°. N-*Acetyl*: m.p. 86–7°. $k = 3{\cdot}8 \times 10^{-4}$ at 25°. N-*Carboethoxyl*: m.p. 114–16°.

β-*Et ester*: m.p. 182°. α-*Amide*: $C_{11}H_{14}O_3N$. MW, 222. Needles from EtOH. M.p. 180–2°. α-*Nitrile*: $C_{11}H_{12}O_2N_2$. MW, 204. Cryst. from EtOH. M.p. 89°. Sol. C_6H_6. Insol. H_2O. α-*Anilide*: m.p. 164–6°. N-*Acetyl*: m.p. 130–2°. N-*Benzoyl*: m.p. 141–3°. N-*Carboethoxyl*: m.p. 106–8°. α-*Hydrazide*: needles from EtOH. M.p. 166°.

Di-Et ester: $C_{13}H_{17}O_4N$. MW, 251. M.p. 75°. N-*Acetyl*: plates or prisms. M.p. 63–4°. B.p. 214–18°/15 mm. decomp. N-*Benzoyl*: m.p. 53–4°. N-*Carboethoxyl*: m.p. 50°.

α-*Amide*: $C_9H_{10}O_3N_2$. MW, 194. M.p. 195°.

Diamide: $C_9H_{11}O_2N_3$. MW, 193. Leaflets from H_2O. M.p. 198–200°.

α-*Nitrile*: $C_9H_8O_2N_2$. MW, 176. Leaflets from EtOH, needles from $CHCl_3$ or C_6H_6. M.p. 184°. Sol. Me_2CO, Et_2O. Mod. sol. boiling EtOH. Insol. C_6H_6. Sols. show violet fluor. β-*Me ester*: $C_{10}H_{10}O_2N_2$. MW, 190. Needles from EtOH. M.p. 106·5°.

β-*Nitrile*: cryst. M.p. 197° decomp.

α-*Anilide*: needles from EtOH. M.p. about 235° decomp. β-*Amide*: m.p. 185°.

N-*Me*: $C_{10}H_{11}O_4N$. MW, 209. Cryst. M.p. 189° decomp.

N-*Et*: $C_{11}H_{13}O_4N$. MW, 223. M.p. 184–6°.

N-*Phenyl*: $C_{15}H_{13}O_4N$. MW, 271. Prisms from MeOH.Aq. M.p. 165–7°.

N-*Benzyl*: $C_{16}H_{15}O_4N$. MW, 285. M.p. 190° decomp. *Di-Me ester*: $C_{18}H_{19}O_4N$. MW, 313. M.p. 82–3°.

N-*Acetyl*: cryst. from MeOH or H_2O. M.p. 214°. $k = 1{\cdot}05 \times 10^{-3}$ at 25°.

N-*Benzoyl*: cryst. from EtOH. M.p. 197°.

α-*Hydrazide*: needles from EtOH. M.p. 200·5°.

Dihydrazide: prisms from EtOH. M.p. 161°. Sol. EtOH. Spar. sol. H_2O. Insol. Et_2O, C_6H_6, pet. ether. *B,2HCl*: m.p. 201°. *Dipicrate*: orange-red plates from H_2O. M.p. 185°.

v. Heyden, D.R.P., 138,207, (*Chem. Zentr.*, 1903, I, 304).
Leonhardt, D.R.P., 126,962, (*Chem. Zentr.*, 1902, I, 82).
Vorländer, *Ber.*, 1902, **35**, 1685.
Vorländer, Mumme, *Ber.*, 1902, **35**, 1699.
Kalle, D.R.P., 206,903, (*Chem. Zentr.*, 1909, I, 807).
Badische, D.R.P., 136,779, (*Chem. Zentr.*, 1902, II, 1351).
Vorländer, v. Schilling, *Ber.*, 1900, **33**, 554.
Houben, *Ber.*, 1913, **46**, 3998.

Phenylglycine-*m*-carboxylic Acid.

Diamide: m.p. 201–2°. Sol. hot H_2O.

α-*Nitrile*: leaflets from 30% EtOH. M.p. 193°. Sol. hot H_2O, EtOH, Me_2CO, C_6H_6.

Houben, Arnold, *Ber.*, 1908, **41**, 1573.
Lumière, Perrin, *Bull. soc. chim.*, 1903, **29**, 966.

Phenylglycine-*p*-carboxylic Acid.

Cryst. from H_2O. M.p. 219–21° decomp. Spar. sol. H_2O.

α-*Amide*: prisms from EtOH. M.p. 251°. Spar. sol. H_2O. Insol. Et_2O, C_6H_6. β-*Et ester*: needles from EtOH. M.p. 142°.

α-*Nitrile*: cryst. powder from Me_2CO. M.p. 177° decomp.

Einhorn, Seuffert, *Ber.*, 1910, **43**, 3001.
Mauthner, Suida, *Monatsh.*, 1890, **11**, 380.
Houben, Arnold, *Ber.*, 1908, **41**, 1572.

Phenylglycollic Acid.

See Mandelic Acid.

Phenylglycollyltropine.

See Homatropine.

Phenylglyoxal (*Benzoylformaldehyde*)

$C_6H_5{\cdot}\underset{\beta}{C}O{\cdot}\underset{\alpha}{C}HO$

$C_8H_6O_2$ MW, 134

Needles + H_2O from H_2O. M.p. 91°. Sol. usual solvents. Sol. about 35 parts H_2O at 20°. Volatile in steam. Alkalis ⟶ mandelic acid. $KMnO_4$ ⟶ benzoic acid. Above m.p. ⟶ anhydrous compound, a deep yellow oil, b.p. 142°/125 mm., 120°/50 mm.

α-*Oxime*: *see* Isonitrosoacetophenone.

Dioxime: *see* Phenylglyoxime.

α-*Di-Me acetal*: b.p. 247–8°, 133–4°/16 mm.

α-*Di-Et acetal*: b.p. 110°/15 mm.

α-*Hydrazone*: needles from C_6H_6. M.p. 120–1°. Mod. sol. EtOH, hot C_6H_6. Spar. sol. H_2O, Et_2O.

α-*Semicarbazone*: pale yellowish leaflets from EtOH. M.p. 217° decomp.

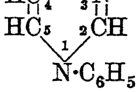
α-*Thiosemicarbazone*: yellowish prisms from EtOH. M.p. 170°. Spar. sol. H_2O, EtOH, Et_2O, $CHCl_3$.

Diphenylhydrazone: *phenylosazone*, yellow leaflets or needles from EtOH. M.p. 152°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

Di-p-*nitrophenylhydrazone*: m.p. 310–11° decomp.

Wieland, Semper, *Ann.*, 1908, **358**, 57.
Russanow, *Ber.*, 1891, **24**, 3501.
Wolff, Lindenhayn, *Ber.*, 1903, **36**, 4127.
Wolff, *Ann.*, 1912, **394**, 34.
Henze, *Z. physiol. Chem.*, 1931, **198**, 84.
Madelung, Oberwegner, *Ber.*, 1932, **65**, 935.
Riley, Gray, *Organic Syntheses*, 1935, XV, 67.

1-Phenylglyoxaline (1-*Phenyliminazole*)



$C_9H_8N_2$ MW, 144

Cryst. M.p. 13°. B.p. 276°. Misc. with most ord. solvents. Insol. H_2O.

B,2HNO_3: prisms. M.p. 82–6°.

B_2,H_2PtCl_6: reddish-yellow leaflets. M.p. 203° decomp. Mod. sol. hot H_2O.

Picrate: yellow needles from EtOH. M.p. 152°. Spar. sol. H_2O, cold EtOH.

Wohl, Marckwald, *Ber.*, 1889, **22**, 576, 1354.
Fischer, Hunsalz, *Ber.*, 1894, **27**, 2206.
Cornforth, Cornforth, *J. Chem. Soc.*, 1947, 102.

2-Phenylglyoxaline (2-*Phenyliminazole*).

Leaflets from C_6H_6, needles from H_2O. M.p. 148–9°. B.p. 340°. Sol. EtOH. Spar. sol. H_2O, C_6H_6.

B,HNO₃: leaflets from EtOH. M.p. 135°.

Acid oxalate: needles from H_2O. M.p. 219°.

Picrate: needles from H_2O. M.p. 238°.

N-*Me*: *see* 1-Methyl-2-phenylglyoxaline.

Fargher, Pyman, *J. Chem. Soc.*, 1919, **115**, 232.
Maquenne, *Ann. chim. phys.*, 1891, **24**, 543.

4-Phenylglyoxaline (4-*Phenyliminazole*).

Leaflets from H_2O. M.p. 128–9°. Very sol. EtOH, Et_2O. Mod. sol. hot H_2O, C_6H_6.

B_2,H_2PtCl_6: orange-red prisms + $3H_2O$. M.p. 215° decomp.

Pinner, *Ber.*, 1902, **35**, 4135.
Clemo, Holmes, Leitch, *J. Chem. Soc.*, 1938, 754.

Phenylglyoxime (*Phenylglyoxal dioxime*)

$C_6H_5{\cdot}C{:}NH{:}O$
$CH{:}N{\cdot}OH$

α-form

$C_8H_8O_2N_2$ MW, 164

Needles from $CHCl_3$ or EtOH.Aq. M.p. 168°. Sol. EtOH, Et_2O, acids. Spar. sol. C_6H_6. Insol. ligroin.

N-*Me*: $C_9H_{10}O_2N_2$. MW, 178. M.p. 209–10°. Sol. AcOH, dil. HCl. Insol. Na_2CO_3. Gives greenish-yellow sol. in amyl alcohol or NaOH.

O:N-*Di-Me*: $C_{10}H_{12}O_2N_2$. MW, 192. M.p. 135°. Sol. 20% HCl.

Dipropionyl: m.p. 75°.

Diphenylurethane: m.p. 136° decomp.

$C_6H_5{\cdot}C{:}N{\cdot}OH$ b
$CH{:}N{\cdot}OH$ a

β-form

Needles from EtOH. M.p. 180°. HCl in Et_2O or AcOH ⟶ α-form.

a-*Me ether*: $C_9H_{10}O_2N_2$. MW, 178. Prisms from ligroin. M.p. 113°. b-*Acetyl*: prisms from pet. ether. M.p. 75°.

Di-Me ether: $C_{10}H_{12}O_2N_2$. MW, 192. Plates from EtOH. M.p. 72°. B.p. 240°/740 mm., 137–8°/40 mm.

Diacetyl: m.p. 71–2°.

Dipropionyl: m.p. 89–90°.

Dibenzoyl: m.p. 150°.

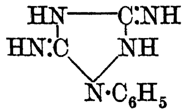
Diphenylurethane: m.p. 146–7° decomp.

Longo, *Gazz. chim. ital.*, 1932, **62**, 139.
Ponzio, *Gazz. chim. ital.*, 1930, **60**, 825.
Avogadro, *Gazz. chim. ital.*, 1926, **56**, 713.
Ponzio, Avogadro, *Gazz. chim. ital.*, 1923, **53**, 25, 311.

Phenylglyoxylic Acid.

See Benzoylformic Acid.

Phenylguanazole



$C_8H_9N_5$ MW, 175

Cryst. M.p. 175°. Sol. EtOH. Mod. sol. $CHCl_3$. Spar. sol. Et_2O, C_6H_6.

B,HCl: needles. M.p. 240°. Sol. H_2O, EtOH.

N-*Nitroso deriv.*: yellow powder. M.p. 245°. Spar. sol. EtOH.

N-*Acetyl deriv.*: prisms from H_2O. M.p. 244°. Sol. warm H_2O, EtOH. Insol. Et_2O.

Diacetyl deriv.: prisms from H_2O. M.p. 212°.

Tetra-acetyl: needles. M.p. 157°.

Pellizzari, Roncagliolo, *Gazz. chim. ital.*, 1901, **31**, i, 477.
Pellizzari, *Gazz. chim. ital.*, 1891, **21**, ii, 146.

Phenylguanidine

$C_6H_5{\cdot}NH{\cdot}C({:}NH){\cdot}NH_2$

$C_7H_9N_3$ MW, 135

Cryst. from C_6H_6. M.p. 66–7°. Sol. EtOH, C_6H_6. Spar. sol. H_2O, Et_2O, CCl_4.

B,HNO₃: needles or prisms. M.p. 128° (118°).

B,HBr: m.p. 71°.

B_2,H_2PtCl_6: m.p. 197–8°.

B_2,H_2SO_4: m.p. 205°. Sol. H_2O. Spar. sol. EtOH.

B_2,H_2CO_3: prisms from H_2O. M.p. 138–40°. Spar. sol. cold H_2O.

Benzoyl deriv.: pale yellow prisms from C_6H_6–pet. ether. M.p. 91–2°. Sol. EtOH, C_6H_6.

Picrate: yellow prisms from EtOH. M.p. 186°.

Dibenzoyl deriv.: needles from EtOH. M.p. 187°.

Picrate: needles. M.p. 223° (218–20°).

Kampf, *Ber.*, 1904, **37**, 1683.
Wheeler, Johnson, *Am. Chem. J.*, 1901, **26**, 417.
Smith, *J. Am. Chem. Soc.*, 1929, **51**, 477.
Schering-Kahlbaum A.-G., D.R.P., 565,881, (*Chem. Zentr.*, 1933, I, 1685).

N-Phenylheptadecylamine.

See Heptadecylaniline.

1-Phenylheptane.

See *n*-Heptylbenzene.

3-Phenylheptane

C_6H_5
$CH_3{\cdot}[CH_2]_3{\cdot}CH{\cdot}CH_2{\cdot}CH_3$

$C_{13}H_{20}$ MW, 176

d-.

B.p. 112°/15 mm. D_4^{25} 0·856. $[\alpha]_D^{25}$ + 0·97°.

dl-.

Obtained by cracking gasoline. B.p. 68–71°/3 mm. n_D^{20} 1·4871.

Levene, Marker, *J. Biol. Chem.*, 1932, **97**, 563.
Tilicheev, Kuruindin, *Chem. Abstracts*, 1931, **25**, 3469.

4-Phenylheptane

$CH_3\cdot[CH_2]_2\cdot CH(C_6H_5)\cdot[CH_2]_2\cdot CH_3$

$C_{13}H_{20}$ MW, 176

B.p. 221–4°/760 mm.

Houston, Kaye, *J. Am. Chem. Soc.*, 1942, **64**, 1576.

Phenylheptanol-1.
See Phenylheptyl Alcohol.

1-Phenylheptanol-5 (7-*Phenylheptanol*-3)

$CH_3\cdot CH_2\cdot CH(OH)\cdot[CH_2]_4\cdot C_6H_5$

$C_{13}H_{20}O$ MW, 192

B.p. 277–8°. D_4^{20} 0·968. n_D^{20} 1·5115.

Zafiriadis, *Compt. rend.*, 1948, **226**, 731.

1-Phenylheptanol-6 (7-*Phenylheptanol*-2)

$CH_3\cdot CH(OH)\cdot[CH_2]_5\cdot C_6H_5$

$C_{13}H_{20}O$ MW, 192

B.p. 169°/50 mm. (164°/23 mm.). D_4^{25} 0·9482 (0·9389). n_D^{25} 1·50575 (1·5028).

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 155.
Davies, Dixon, Jones, *J. Chem. Soc.*, 1930, 471.

2-Phenylheptanol-3

$CH_3\cdot[CH_2]_3\cdot CH(OH)\cdot CH(C_6H_5)\cdot CH_3$

$C_{13}H_{20}O$ MW, 192

B.p. 156–60°/30 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

3-Phenylheptanol-4

$CH_3\cdot[CH_2]_2\cdot CH(OH)\cdot CH(C_6H_5)\cdot CH_2\cdot CH_3$

$C_{13}H_{20}O$ MW, 192

B.p. 145–7°/20 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

4-Phenylheptanol-4 (*Dipropylphenylcarbinol, ω-diethylbenzyl alcohol*)

$CH_3\cdot CH_2\cdot CH_2\cdot C(OH)(C_6H_5)\cdot CH_2\cdot CH_2\cdot CH_3$

$C_{13}H_{20}O$ MW, 192

B.p. 134°/26 mm. D^0 0·9589, D^{15} 0·9470. n_D 1·516.
Acetyl: b.p. 160°/19 mm. D^{15} 0·8973.

Amouroux, Murat, *Compt. rend.*, 1912, **154**, 993.
Gilman, Fothergill, Parker, *Rec. trav. chim.*, 1929, **48**, 748.

2-Phenylheptanone-3

$CH_3\cdot[CH_2]_3\cdot CO\cdot CH(C_6H_5)\cdot CH_3$

$C_{13}H_{18}O$ MW, 190

B.p. 256–7°.
Semicarbazone: m.p. 129°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

2-Phenylheptanone-6

$CH_3\cdot CO\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH(C_6H_5)\cdot CH_3$

$C_{13}H_{18}O$ MW, 190

B.p. 136–40°/13 mm. D_4^{20} 0·9629.
p-*Nitrophenylhydrazone*: m.p. 117°.

Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 270.

3-Phenylheptanone-2

$CH_3\cdot[CH_2]_3\cdot CH(C_6H_5)\cdot CO\cdot CH_3$

$C_{13}H_{18}O$ MW, 190

B.p. 250–1°. D_4^0 0·960.
Oxime: m.p. 63–6°.
Semicarbazone: m.p. 156–8°.

Tiffeneau, Lévy, Jullien, *Bull. soc. chim.*, 1931, **49**, 1788.

3-Phenylheptanone-4

$CH_3\cdot CH_2\cdot CH_2\cdot CO\cdot CH(C_6H_5)\cdot CH_2\cdot CH_3$

$C_{13}H_{18}O$ MW, 190

B.p. 242–5°.
Semicarbazone: m.p. 106–7°.

Tiffeneau, Lévy, Jullien, *Bull. soc. chim.*, 1931, **49**, 1788.

4-Phenylheptanone-3

$CH_3\cdot CH_2\cdot CH_2\cdot CH(C_6H_5)\cdot CO\cdot CH_2\cdot CH_3$

$C_{13}H_{18}O$ MW, 190

B.p. 240–5°.
Semicarbazone: m.p. 108–9°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

4-Phenyl-3-heptene (1-*Propyl*-1-*phenylbutylene*-1, 4-*phenyl*-3-*heptylene*)

$CH_3\cdot CH_2\cdot CH_2\cdot C(C_6H_5){:}CH\cdot CH_2\cdot CH_3$

$C_{13}H_{18}$ MW, 174

B.p. 228°/760 mm. D^{25} 0·8855. n_D^{15} 1·522.

Amouroux, Murat, *Compt. rend.*, 1912, **154**, 993.

1-Phenylheptyl Alcohol (n-*Hexylphenylcarbinol*, 1-*phenylheptanol*-1)

$CH_3\cdot[CH_2]_5\cdot CH(OH)\cdot C_6H_5$

$C_{13}H_{20}O$ MW, 192

B.p. 275°, 176°/40 mm., 156°/25 mm.
Phenylurethane: m.p. 77°.

Colacicchi, *Atti accad. Lincei*, 1910, **19**, ii, 601.

3-Phenylheptyl Alcohol (*3-Phenylheptanol-1*)

$$CH_3\cdot[CH_2]_3\cdot CH(C_6H_5)\cdot CH_2\cdot CH_2OH$$

$C_{13}H_{20}O$ MW, 192

l-.
B.p. 150°/10 mm. D_4^{27} 0·947. $[\alpha]_D^{27}$ − 1·45°.
dl-.
B.p. 116–20°/3 mm. D_{25}^{25} 0·9466. n_D^{20} 1·5070.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2525.
Levene, Marker, *J. Biol. Chem.*, 1932, **97**, 563.

5-Phenylheptyl Alcohol (*5-Phenylheptanol-1*)

$$CH_3\cdot CH_2\cdot CH(C_6H_5)\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH_2OH$$

$C_{13}H_{20}O$ MW, 192

d-.
B.p. 145°/1 mm. D_4^{25} 0·952. $[\alpha]_D^{25}$ + 1·47°.
l-.
B.p. 123°/1 mm.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 749; 1935, **110**, 329.

7-Phenylheptyl Alcohol (*7-Phenylheptanol-1*)

$$C_6H_5\cdot[CH_2]_6\cdot CH_2OH$$

$C_{13}H_{20}O$ MW, 192

Liq. with faint odour of roses. B.p. 170–2°/15 mm.
Acetyl: b.p. 188–90°/24 mm.

v. Braun, *Ber.*, 1911, **44**, 2878.

Phenylheptylene.
See Phenylheptene.

1-Phenyl-1 : 3-hexadiene (*1-Styryl-1-butylene*)

$$CH_3\cdot CH_2\cdot CH{:}CH\cdot CH{:}CH\cdot C_6H_5$$

$C_{12}H_{14}$ MW, 158

Oil B.p. 128°/16 mm. D_4^{12} 0·9253. n_D^{12} 1·60252. Na + EtOH ⟶ 1-phenyl-2-hexene.

Klages, *Ber.*, 1907, **40**, 1770.
Emerson, *J. Org. Chem.*, 1945, **10**, 464.

6-Phenyl-1 : 3-hexadiene

$$C_6H_5\cdot CH_2\cdot CH_2\cdot CH{:}CH\cdot CH{:}CH_2$$

$C_{12}H_{14}$ MW, 158

B.p. 99·5–100·5°/11 mm. D_4^{13} 0·9304. n_D^{13} 1·5446.

Cohen, *J. Chem. Soc.*, 1935, 433.
Paul, Tchelitcheff, *Bull. soc. chim.*, 1948, 108.

1-Phenyl-1 : 5-hexadiene (*4-Styryl-1-butylene*)

$$CH_2{:}CH\cdot CH_2\cdot CH_2\cdot CH{:}CH\cdot C_6H_5$$

$C_{12}H_{14}$ MW, 158

B.p. 102·5–103·5°/8 mm. D_4^{25} 0·9005. n_D^{25} 1·5421.

Levy, Cope, *J. Am. Chem. Soc.*, 1944, **66**, 1686.

3-Phenyl-1 : 5-hexadiene

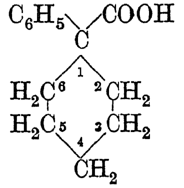
$$CH_2{:}CH\cdot CH_2\cdot CH(C_6H_5)\cdot CH{:}CH_2$$

$C_{12}H_{14}$ MW, 158

B.p. 78°/8 mm. D_4^{25} 0·8911. n_D^{25} 1·5141. Heat ⟶ 1-phenyl-1 : 5-hexadiene.

Levy, Cope, *J. Am. Chem. Soc.*, 1944, **66**, 1685.

1-Phenylhexahydrobenzoic Acid (*1-Phenylcyclohexane-1-carboxylic acid*)



$C_{13}H_{16}O_2$ MW, 204

Cryst. M.p. 123–4°.
Et ester: $C_{15}H_{20}O_2$. B.p. 105–10°/0·1 mm.
Amide: $C_{13}H_{17}ON$. MW, 203. Cryst. from C_6H_6–pet. ether. M.p. 95–6°.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.
Rubin, Wishinski, *J. Am. Chem. Soc.*, 1946, **68**, 830.

2-Phenylhexahydrobenzoic Acid (*2-Phenylcyclohexane-1-carboxylic acid*).

Prisms from pet. ether. M.p. 105–7° (104–5°). Sol. MeOH, Et_2O, Me_2CO, $CHCl_3$, warm pet. ether. Spar. sol. H_2O.

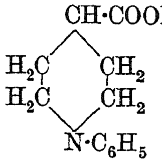
Kipping, Perkin, *J. Chem. Soc.*, 1890, **57**, 316.
Ranedo, Léon, *Chem. Abstracts*, 1927, **21**, 1108.
Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 70.
Gutsche, Johnson, *J. Am. Chem. Soc.*, 1946, **68**, 2242.

4-Phenylhexahydrobenzoic Acid (*4-Phenylcyclohexane-1-carboxylic acid*).

Exists in two forms.
(i) Leaflets from 50% AcOH. M.p. 204° (202°). Fuming HCl at 170° ⟶ (ii).
Me ester: $C_{14}H_{18}O_2$. MW, 218. M.p. 28°. B.p. 172–3°/15 mm.
(ii) Needles from 40% EtOH. M.p. 113°. Sol. EtOH, $CHCl_3$, AcOH. Less sol. Et_2O, pet. ether. Sol. 1000 parts boiling H_2O. Conc. HCl at 180° ⟶ (i).

Rassow, *Ann.*, 1894, **282**, 147.
Nenitzescu, Gavát, *Ann.*, 1935, **519**, 266.
Johnson, Offenhauer, *J. Am. Chem. Soc.*, 1945, **67**, 1047.

N-Phenylhexahydroisonicotinic Acid (*N-Phenylpiperidine-4-carboxylic acid, N-phenylisonipecotic acid*)



$C_{12}H_{15}O_2N$ MW, 205

Cryst. from EtOH. M.p. 131°.

B,HBr: cryst. from H_2O. M.p. 218–19°.

Me ester: $C_{13}H_{17}O_2N$. MW, 219. Cryst. from ligroin. M.p. 46°.

Anilide: m.p. 210°.

Methiodide: decomp. 180°.

Picrate: cryst. from MeOH. M.p. 207° decomp.

Prelog, Hanousek, *Chem. Abstracts*, 1934, **28**, 5825.

Phenylhexahydrophenol.

See Phenylcyclohexanol.

Phenyl hexahydrostyryl Ketone.

See Hexahydrobenzylideneacetophenone.

1-Phenylhexane (*n-Hexylbenzene*)

$CH_3{\cdot}[CH_2]_4{\cdot}CH_2{\cdot}C_6H_5$

$C_{12}H_{18}$ MW, 162

B.p. 219–20° (215°). D_4^{20} 0·8613. n_D^{20} 1·490.

v. Braun, Deutsch, *Ber.*, 1912, **45**, 2180.

Sabatier, Mailhe, *Compt. rend.*, 1914, **158**, 834.

Gilman, Meals, *J. Org. Chem.*, 1943, **8**, 126.

2-Phenylhexane (*ω-Methylbutyltoluene*)

C_6H_5
$CH_3{\cdot}[CH_2]_3{\cdot}CH{\cdot}CH_3$

$C_{12}H_{18}$ MW, 162

d-.

B.p. 100°/22 mm. D_4^{25} 0·855. $[\alpha]_D^{25}$ + 1·96°.

dl-.

B.p. 208°/760 mm. D_4^{15} 0·869. n^{15} 1·492.

Brochet, *Bull. soc. chim.*, 1893, **9**, 687.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 749.

Gilman, Meals, *J. Org. Chem.*, 1943, **8**, 126.

Huston, Kaye, *J. Am. Chem. Soc.*, 1942, **64**, 1576.

Spiegler, Tinker, *J. Am. Chem. Soc.*, 1939, **61**, 1002.

3-Phenylhexane (*ω-Ethylpropyltoluene*)

C_6H_5
$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{\cdot}CH_2{\cdot}CH_3$

$C_{12}H_{18}$ MW, 162

d-.

B.p. 103°/25 mm. D_4^{24} 0·863. $[\alpha]_D^{24}$ + 0·57°.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 749.

Spiegler, Tinker, *J. Am. Chem. Soc.*, 1939, **61**, 1002.

Phenylhexanol-1.

See Phenylhexyl Alcohol.

1-Phenylhexanol-3 (*Propyl-β-phenylethylcarbinol*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$

$C_{12}H_{18}O$ MW, 178

d-.

Needles. M.p. 34°. B.p. 146°/16 mm. Volatile in steam.

Acid phthalate: m.p. 75°. $[\alpha]_{5893}$ + 25·5°.

Brucine salt: cryst. from Me_2CO. M.p. 75°. $[\alpha]_{5893}$ − 12·5°.

l-.

Needles. M.p. 34°. B.p. 146°/16 mm. Volatile in steam.

Acid phthalate: m.p. 75°. $[\alpha]_{5893}$ − 25·5°.

Brucine salt: cryst. from Me_2CO. M.p. 95°. $[\alpha]_{5893}$ − 2·1° in EtOH.

Formyl: b.p. 147°/18 mm. D_4^{20} 0·9872.

Acetyl: b.p. 154°/20 mm. D_4^{20} 0·9725.

dl.-

Acid phthalate: prisms from EtOH. M.p. 108°.

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1094.

1-Phenylhexanol-5 (*6-Phenylhexanol-2*)

$CH_3{\cdot}CH(OH){\cdot}[CH_2]_4{\cdot}C_6H_5$

$C_{12}H_{18}O$ MW, 178

B.p. 148°/18 mm. D_4^{20} 0·9567. n_D^{20} 1·50787.

Phenylurethane: needles from pet. ether. M.p. 65°.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

2-Phenylhexanol-2 (*Methyl-n-butylphenylcarbinol*)

C_6H_5
$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}C(OH){\cdot}CH_3$

$C_{12}H_{18}O$ MW, 178

B.p. 129–30°/4 mm. D^{20} 0·9522. n_D^{20} 1·5091.

Conant, Carlson, *J. Am. Chem. Soc.*, 1932, **54**, 4055.

Gilman, Meals, *J. Org. Chem.*, 1943, **8**, 126.

2-Phenylhexanol-3 (*Propyl-α-phenylethylcarbinol*)

C_6H_5
$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH{\cdot}CH_3$

$C_{12}H_{18}O$ MW, 178

B.p. 150–5°/30 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

3-Phenylhexanol-3 (*Ethyl-n-propylphenylcarbinol*)

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}\overset{C_6H_5}{\overset{|}{C}}(OH){\cdot}CH_2{\cdot}CH_3$$

$C_{12}H_{18}O$ MW, 178

B.p. 134°/27 mm. n_D^{20} 1·5100. D_{20}^{20} 0·966.

Gilman, Meals, *J. Org. Chem.*, 1943, **8**, 126.

3-Phenylhexanol-4 (*Ethyl-α-phenylpropylcarbinol*)

$$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}\overset{C_6H_5}{\overset{|}{C}H}{\cdot}CH_2{\cdot}CH_3$$

$C_{12}H_{18}O$ MW, 178

α-.
M.p. 47·5°.
Phenylurethane : m.p. 109°.

β-.
B.p. 117–125°/15 mm., 130°/20 mm.
Phenylurethane : m.p. 127°.

Jullien, Kayser, *Bull. soc. chim.*, 1937, **4**, 704.

1-Phenylhexanone-3 (*Propyl β-phenylethyl ketone*)

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$$

$C_{12}H_{16}O$ MW, 176

B.p. 133–5°/8 mm.
Oxime : m.p. 43°.
Semicarbazone : cryst. from C_6H_6. M.p. 79°.

Rupe, Hirschmann, *Helv. Chim. Acta*, 1931, **14**, 698.

1-Phenylhexanone-4 (6-*Phenylhexanone*-3)

$$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$$

$C_{12}H_{16}O$ MW, 176

Colourless oil. B.p. 137–40°/10 mm.
Semicarbazone : cryst. from EtOH. M.p. 153–4° (149°).

Rupe, Hirschmann, *Helv. Chim. Acta*, 1931, **14**, 700.
Tiffeneau, Deux, *Compt. rend.*, 1942, **214**, 894.

1-Phenylhexanone-5 (6-*Phenylhexanone*-2)

$$CH_3{\cdot}CO{\cdot}[CH_2]_4{\cdot}C_6H_5$$

$C_{12}H_{16}O$ MW, 176

B.p. 137°/8 mm.
Semicarbazone : m.p. 143°.

Ramart-Lucas, Labaune, *Ann. chim.*, 1931, **16**, 276.
Van Veen, *Rec. trav. chim.*, 1939, **58**, 521.

2-Phenylhexanone-3 (*Propyl α-phenylethyl ketone*)

$$CH_3{\cdot}[CH_2]_2{\cdot}CO{\cdot}\overset{C_6H_5}{\overset{|}{C}H}{\cdot}CH_3$$

$C_{12}H_{16}O$ MW, 176

B.p. 240–2°.
Semicarbazone : m.p. 148–9°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

2-Phenylhexanone-5 (5-*Phenylhexanone*-2)

$$CH_3{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}\overset{C_6H_5}{\overset{|}{C}H}{\cdot}CH_3$$

$C_{12}H_{16}O$ MW, 176

B.p. 122–5°/9 mm.
Semicarbazone : m.p. 147°.

Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 270.

3-Phenylhexanone-2

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}\overset{C_6H_5}{\overset{|}{C}H}{\cdot}CO{\cdot}CH_3$$

$C_{12}H_{16}O$ MW, 176

B.p. 235–6°, 114–15°/13 mm. D_4^0 0·970. n_D^{20} 1·5020.
Oxime : m.p. 42–3°.
Semicarbazone : m.p. 137–137·5°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.
Suter, Weston, *J. Am. Chem. Soc.*, 1942, **64**, 533.
Deux, *Compt. rend.*, 1941, **213**, 211.

3-Phenylhexanone-4 (4-*Phenylhexanone*-3)

$$CH_3{\cdot}CH_2{\cdot}CO{\cdot}\overset{C_6H_5}{\overset{|}{C}H}{\cdot}CH_2{\cdot}CH_3$$

$C_{12}H_{16}O$ MW, 176

Yellow oil. B.p. 114–16°/13 mm. D^{20} 0·965, D^0 0·978.
Oxime : m.p. 57–8°.
Semicarbazone : m.p. 139–40°. Mod. sol. EtOH, C_6H_6.

Tiffeneau, Lévy, *Bull. soc. chim.*, 1923, **33**, 735.
Jullien, Kayser, *Bull. soc. chim.*, 1937, **4**, 705.

6-Phenyl-1-hexene

$$C_6H_5{\cdot}[CH_2]_3{\cdot}CH_2{\cdot}CH{:}CH_2$$

$C_{12}H_{16}$ MW, 160

B.p. 94–5°/10 mm. D_4^{20} 0·8839. n_D^{20} 1·5033.

v. Braun, Deutsch, Schmatlock, *Ber.*, 1912, **45**, 1257.
Kozacik, Reid, *J. Am. Chem. Soc.*, 1938, **60**, 2436.

1-Phenyl-2-hexene (sym.-*Propylbenzylethylene*, 1-*propyl*-3-*phenylpropylene*)

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH_2{\cdot}C_6H_5$$

$C_{12}H_{16}$ MW, 160

B.p. 108°/16 mm. D_4^{16} 0·8898. n_D^{16} 1·5058.

Klages, *Ber.*, 1907, **40**, 1771.

2-Phenyl-2-hexene

$$\begin{array}{c} \qquad\qquad\qquad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}C{\cdot}CH_3 \end{array}$$

$C_{12}H_{16}$ MW, 160

B.p. 66–8°/1 mm. D_4^{20} 0·910. n_D^{20} 1·5200.

Nazarov, Kotlyarevskiĭ, *Chem. Abstracts*, 1949, **43**, 118.

1-Phenyl-3-hexene

$$CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$$

$C_{12}H_{16}$ MW, 160

B.p. 222°. D_{20}^{20} 0·8806. n_D^{20} 1·5039.

Treibs, *Ber.*, 1947, **80**, 97.

2-Phenyl-3-hexene

$$\begin{array}{c} \qquad\qquad\qquad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH{\cdot}CH_3 \end{array}$$

$C_{12}H_{16}$ MW, 160

B.p. 84°/10 mm.

Riiber, *Ber.*, 1903, **36**, 1405.

1-Phenyl-1-hexenone-3

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH{:}CH{\cdot}C_6H_5$$

$C_{12}H_{14}O$ MW, 174

Phenylhydrazone: exists in 2 forms. α-: yellow cryst. M.p. 99—100°. Phototropic. γ-: m.p. 86°.

Gheorghui, Matei, *Bull. soc. chim.*, 1939, **6**, 1324.

1-Phenyl-1-hexenone-5

$$CH_3{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}CH{\cdot}C_6H_5$$

$C_{12}H_{14}O$ MW, 174

B.p. 153–5°/10 mm., 97–9°/0·3 mm. D_{25}^{25} 0·9997. n_D^{25} 1·5458.

Semicarbazone: cryst. from AcOEt. M.p. 132°.

Fischer, Wiedemann, *Ann.*, 1935, **520**, 69.

Kimel, Cope, *J. Am. Chem. Soc.*, 1943, **65**, 1992.

3-Phenyl-1-hexenone-4

$$\begin{array}{c} \qquad\quad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH{\cdot}CH{:}CH_2 \end{array}$$

$C_{12}H_{14}O$ MW, 174

B.p. 121–2°/15 mm.

Oxime: m.p. 99°.

Semicarbazone: m.p. 148°.

Deux, *Compt. rend.*, 1939, **208**, 1090; 1943, **216**, 776.

3-Phenyl-1-hexenone-5

$$\begin{array}{c} \qquad\quad C_6H_5 \\ CH_3{\cdot}CO{\cdot}CH_2{\cdot}CH{\cdot}CH{:}CH_2 \end{array}$$

$C_{12}H_{14}O$ MW, 174

B.p. 85–6°/1 mm. D_{25}^{25} 0·9877. n_D^{25} 1·5193.

Kimel, Cope, *J. Am. Chem. Soc.*, 1943, **65**, 1992.

2-Phenyl-2-hexenone-4

$$\begin{array}{c} \qquad\qquad\qquad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH{:}C{\cdot}CH_3 \end{array}$$

$C_{12}H_{14}O$ MW, 174

B.p. 175–80°/47 mm. D_{20}^{20} 0·9976. n_D^{23} 1·4999.

Chelintsev, Pataraya, *Chem. Abstracts*, 1941, **35**, 6571.

3-Phenyl-2-hexenone-5

$$\begin{array}{c} \qquad\qquad C_6H_5 \\ CH_3{\cdot}CO{\cdot}CH_2{\cdot}C{:}CH{\cdot}CH_3 \end{array}$$

$C_{12}H_{14}O$ MW, 174

B.p. 138°/14 mm.

Semicarbazone: plates from EtOH. M.p. 185°.

Johnson, Kon, *J. Chem. Soc.*, 1926, 2758.

3-Phenyl-3-hexenone-5

$$\begin{array}{c} \qquad C_6H_5 \\ CH_3{\cdot}CO{\cdot}CH{:}C{\cdot}CH_2{\cdot}CH_3 \end{array}$$

$C_{12}H_{14}O$ MW, 174

B.p. 138°/14 mm.

Semicarbazone: plates. M.p. 158°.

Johnson, Kon, *J. Chem. Soc.*, 1926, 2758.

1-Phenylhexyl Alcohol (1-*Phenylhexanol*-1, n-*amylphenylcarbinol*)

$$CH_3{\cdot}[CH_2]_4{\cdot}CH(OH){\cdot}C_6H_5$$

$C_{12}H_{18}O$ MW, 178

Viscous liq. B.p. 170°/50 mm., 128°/5 mm. D_4^{25} 0·9477. n_D^{25} 1·5042, n_D^{20} 1·5105.

Davies, Dixon, Jones, *J. Chem. Soc.*, 1930, 470.

Ipatieff, Haensel, *J. Am. Chem. Soc.*, 1942, **64**, 520.

2-Phenylhexyl Alcohol (2-*Phenylhexanol*-1)

$$\begin{array}{c} \qquad\qquad C_6H_5 \\ CH_3{\cdot}[CH_2]_3{\cdot}CH{\cdot}CH_2OH \end{array}$$

$C_{12}H_{18}O$ MW, 178

d-.

B.p. 127°/1 mm. D_4^{25} 0·967. $[\alpha]_D^{25}$ + 1·96°.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 772.

3-Phenylhexyl Alcohol (3-*Phenylhexanol*-1)

$$\begin{array}{c} \qquad C_6H_5 \\ CH_3{\cdot}[CH_2]_2{\cdot}CH{\cdot}CH_2{\cdot}CH_2OH \end{array}$$

$C_{12}H_{18}O$ MW, 178

d-.

B.p. 127°/5 mm. D_4^{25} 0·955. n_D^{25} 1·5101. $[\alpha]_D^{25}$ + 2·51°.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 765.

4-Phenylhexyl Alcohol (4-*Phenylhexanol*-1)

$$\begin{array}{c} C_6H_5 \qquad\qquad \\ CH_3{\cdot}CH_2{\cdot}CH{\cdot}[CH_2]_2{\cdot}CH_2OH \end{array}$$

$C_{12}H_{18}O$ MW, 178

d-.
B.p. 125°/5 mm. $[\alpha]_D^{25}$ + 0·21°.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 335.

6-Phenylhexyl Alcohol (*6-Phenylhexanol-1*)

$C_6H_5\cdot[CH_2]_5\cdot CH_2OH$

$C_{12}H_{18}O$ MW, 178

B.p. 160–1°/13 mm.
Me ether: $C_{13}H_{20}O$. MW, 192. B.p. 140°/13 mm.
Acetyl: b.p. 166–8°/13 mm.

v. Braun, *Ber.*, 1911, **44**, 2876.
v. Braun, Deutsch, *Ber.*, 1912, **45**, 2177.

1-Phenylhippuric Acid (*1-Benzoylamino-phenylacetic acid*)

C_6H_5
$C_6H_5\cdot CO\cdot NH\cdot CH\cdot COOH$

$C_{15}H_{13}O_3N$ MW, 255

Needles from EtOH. M.p. 175·5°. Sol. EtOH, Et_2O, hot H_2O.
Et ester: $C_{17}H_{17}O_3N$. MW, 283. Cryst. M.p. 84°.
Phenyl ester: $C_{21}H_{17}O_3N$. MW, 331. Needles from EtOH. M.p. 131°.

Bayer, D.R.P., 55,026.
Kossel, *Ber.*, 1891, **24**, 4151.

***N*-Phenylhippuric Acid.**
See under Phenylglycine.

1-Phenylhydantoic Acid (*α-Ureido-phenyl-acetic acid*)

C_6H_5
$H_2N\cdot CO\cdot NH\cdot CH\cdot COOH$

$C_9H_{10}O_3N_2$ MW, 194

Prisms from H_2O. M.p. 196–196·5° decomp. (178°). Spar. sol. cold H_2O.
Et ester: $C_{11}H_{14}O_3N_2$. MW, 222. Cryst. M.p. 139–40°. Sol. EtOH, Et_2O, hot H_2O.
Amide: $C_9H_{11}O_2N_3$. MW, 193. Prisms from EtOH.Aq. M.p. 223° decomp. Sol. EtOH, hot H_2O. Less sol. Et_2O, C_6H_6.

Pinner, *Ber.*, 1888, **21**, 2326.
Dakin, Dudley, *J. Biol. Chem.*, 1914, **18**, 49.
Pinner, Spilker, *Ber.*, 1889, **22**, 697.
Kossel, *Ber.*, 1891, **24**, 4150.

4-Phenylhydantoic Acid

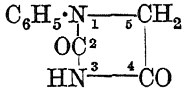
$C_6H_5\cdot NH\cdot CO\cdot NH\cdot CH_2\cdot COOH$

$C_9H_{10}O_3N_2$ MW, 194

Needles. M.p. 197° decomp.
Et ester: needles. M.p. 108–9°.

Bailey, *Am. Chem. J.*, 1902, **28**, 386.
Pressman, Bryden, Pauling, *J. Am. Chem. Soc.*, 1948, **70**, 1352.

1-Phenylhydantoin



$C_9H_8O_2N_2$ MW, 176

Fine needles. M.p. 193–4°. Mod. sol. EtOH. Spar. sol. H_2O.

Schwebel, *Ber.*, 1877, **10**, 2049.
Friedrich, Beckurts, *Arch. Pharm.*, 1899, **237**, 337.

3-Phenylhydantoin.
Needles from H_2O. M.p. 159–60°. Sol. EtOH, Me_2CO, hot C_6H_6, conc. min. acids. Spar. sol. Et_2O.

Mouneyrat, *Ber.*, 1900, **33**, 2394.

5-Phenylhydantoin.
Needles. M.p. 179°. Sol. EtOH, alkalis. Spar. sol. H_2O.
N-*Me*: $C_{10}H_{10}O_2N_2$. MW, 190. Needles from H_2O. M.p. 161°. Spar. sol. cold EtOH.
N-*Et*: prisms from H_2O. M.p. 94°. Very sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O.
N-*Acetyl*: needles from C_6H_6. M.p. 145°.

Pinner, Lifschütz, *Ber.*, 1887, **20**, 2355.
Pinner, *Ber.*, 1888, **21**, 2325.
Lehmann, *Ber.*, 1901, **34**, 372.
Bergs, D.R.P., 566,094, (*Chem. Abstracts*, 1933, **27**, 1001).
Fischer, Ekeley, Ronzio, *J. Am. Chem. Soc.*, 1942, **64**, 1434.

1-Phenylhydracrylic Acid.
See Tropic Acid.

2-Phenylhydracrylic Acid.
See β-Hydroxyhydrocinnamic Acid.

α-Phenylhydratropic Acid.
See 1 : 1-Diphenylpropionic Acid.

Phenylhydrazidine (*Acetylamidrazone*)

$CH_3\cdot CO\cdot C(NH_2)\colon N\cdot NH\cdot C_6H_5$
γ β α

$C_9H_{11}ON_3$ MW, 177

Yellow needles. M.p. 183°. Spar. sol. C_6H_6, ligroin, boiling H_2O.
γ-N-*Acetyl*: needles. M.p. 143°. Very sol. $CHCl_3$. Mod. sol. Et_2O. Insol. ligroin.
Phenylhydrazone: cryst. powder or needles. M.p. 224°. Sol. Et_2O, $CHCl_3$, Me_2CO. *B,HNO*$_2$: needles. M.p. 135°.

Bamberger, Lorenzen, *Ber.*, 1892, **25**, 3541.
Bamberger, Gruyter, *Ber.*, 1893, **26**, 2785; *J. prakt. Chem.*, 1901, **64**, 234.

Phenylhydrazine (*Hydrazinobenzene*)

$C_6H_5\cdot NH\cdot NH_2'$

$C_6H_8N_2$ MW, 108

Plates or prisms. M.p. 23°. B.p. 241–2°, 137–8°/18 mm. $D_4^{20\cdot 3}$ 1·0978. $n_D^{20\cdot 3}$ 1·60813. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol.

H_2O, ligroin. Heat of comb. C_v 879·2 Cal. $k = 1{\cdot}62 \times 10^{-9}$ at 15°. Weak base.

B,HF: leaflets. M.p. 166–7° decomp.

B,HCl: leaflets from EtOH. M.p. 240°.

B,HNO_3: leaflets. M.p. 145°.

N′-*Phenyl*: *see* Hydrazobenzene.

N′-*Formyl*: leaflets from EtOH. M.p. 145°. Sol. hot H_2O, EtOH. Spar. sol. cold H_2O, $CHCl_3$, C_6H_6.

N-*Acetyl*: *N*-phenylacethydrazide. Leaflets from C_6H_6–ligroin. M.p. 125–6°. Sol. EtOH, $CHCl_3$, C_6H_6. Spar. sol. Et_2O, ligroin. N′-*Formyl*: cryst. M.p. 77–8°.

N′-*Acetyl*: *N′*-phenylacethydrazide. Prisms. M.p. 129°. Sol. hot H_2O, EtOH. Spar. sol. cold H_2O, Et_2O. Reduces Fehling's. N-*Formyl*: cryst. M.p. 86°.

N : N′-*Diacetyl*: plates or needles from EtOH–C_6H_6. M.p. 107–8°. Very sol. boiling H_2O, EtOH, $CHCl_3$. Spar. sol. Et_2O. Reduces cold Fehling's.

N′-*Propionyl*: plates from $CHCl_3$. M.p. 157–8°.

N′-*Butyryl*: plates from H_2O. M.p. 103–4°.

N-*Isobutyryl*: plates or needles from pet. ether. M.p. 46–8°.

N′-*Isobutyryl*: leaflets from H_2O. M.p. 142–3°.

N′-*Isovaleryl*: leaflets from 30% EtOH. M.p. 112–112·5°.

N-*Benzoyl*: needles from H_2O or EtOH.Aq. M.p. 70°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O. Reduces warm Fehling's.

N′-*Benzoyl*: prisms from EtOH. M.p. 168°. Mod. sol. hot EtOH, C_6H_6. Spar. sol. H_2O, Et_2O.

N : N′-*Dibenzoyl*: prisms from EtOH. M.p. 177–8°. Mod. sol. hot EtOH. Spar. sol. H_2O. Reduces $NH_3.AgNO_3$.

N-*Nitroso*: $C_6H_5N(NO){\cdot}NH_2$. $C_6H_7ON_3$. MW, 137. Yellow cryst. M.p. 51°. Dil. HCl ⟶ phenyl azide. Poisonous. Forms red Cu salt.

Michaelis, Schmidt, *Ann.*, 1889, **252**, 302.
Leighton, *Am. Chem. J.*, 1898, **20**, 677.
Widman, *Ber.*, 1894, **27**, 2964.
Thompson, *J. Soc. Dyers Colourists*, 1921, **37**, 7.
Coleman, *Organic Syntheses*, Collective Vol. I, 432.
Wurster, *Ber.*, 1887, **20**, 2633.
Bamberger, Hauser, *Ann.*, 1910, **375**, 317.

Phenylhydrazine-α-carboxylic Acid (*α-Phenylhydrazinoformic acid*)

$C_6H_5{\cdot}N(NH_2){\cdot}COOH$

$C_7H_8O_2N_2$ MW, 152

Me ester: $C_8H_{10}O_2N_2$. MW, 166. Rhombic prisms from pet. ether. M.p. 69–70°.

Et ester: $C_9H_{12}O_2N_2$. MW, 180. Plates. M.p. 24–5°. B.p. 157°/15 mm. Spar. sol. usual solvents. Alc. $FeCl_3$ ⟶ red col. *B,HCl*: needles. M.p. 157–8°.

Amide: *see* 2-Phenylsemicarbazide.

Rupe, Gebhardt, *Ber.*, 1899, **32**, 11.
Busch, Limpach, *Ber.*, 1911, **44**, 1583.
Willstätter, Ulbrich, Pogány, Maimeri, *Ann.*, 1929, **477**, 168.

Phenylhydrazine-β-carboxylic Acid (*ω-Phenylcarbazinic acid, β-phenylhydrazinoformic acid*)

$C_6H_5{\cdot}NH{\cdot}NH{\cdot}COOH$

$C_7H_8O_2N_2$ MW, 152

Free acid not isolated.

K salt: needles from EtOH. M.p. 243° decomp. Sol. H_2O, EtOH.

Me ester: $C_8H_{10}O_2N_2$. MW, 166. Prisms from H_2O. M.p. 115–17°.

Et ester: $C_9H_{12}O_2N_2$. MW, 180. Cryst. from EtOH.Aq. M.p. 82–3°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6, hot H_2O. Conc. H_2SO_4 sol. with $FeCl_3$ ⟶ intense red col.

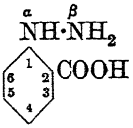
Phenyl ester: $C_{13}H_{12}O_2N_2$. MW, 228. Needles. M.p. 122–3°.

Amide: *see* 1-Phenylsemicarbazide.

B,$C_6H_5{\cdot}NH{\cdot}NH_2$: cryst. powder. M.p. 80° decomp.

Busch, Limpach, *Ber.*, 1911, **44**, 1582.
Freundler, *Bull. soc. chim.*, 1901, **25**, 859.
Busch, Stern, *J. prakt. Chem.*, 1899, **60**, 236.
Heller, *Ann.*, 1891, **263**, 281.

Phenylhydrazine-*o*-carboxylic Acid (*o-Hydrazinobenzoic acid*)



$C_7H_8O_2N_2$ MW, 152

Needles from H_2O. M.p. 249°. Mod. sol. hot H_2O. Spar. sol. EtOH, Et_2O. Reduces Fehling's and $NH_3.AgNO_3$ in the cold.

B,HCl: m.p. 189–90°.

B,HBr: needles. M.p. 207–10°.

Nitrile: *o*-cyanophenylhydrazine. $C_7H_7N_3$. MW, 133. Leaflets from H_2O. M.p. 152–3°. Sol. EtOH, Et_2O. Insol. ligroin. *B,HCl*: needles. M.p. 160–1°. *B_2,H_2SO_4*: m.p. 215–16°. *Picrate*: needles. M.p. 238°.

α-N-*Me*: $C_8H_{10}O_2N_2$. MW, 166. Cryst. from ligroin. M.p. 120°. Very sol. EtOH.

Fischer, *Ber.*, 1880, **13**, 680.
Acree, *Am. Chem. J.*, 1907, **37**, 365.
Gabriel, *Ber.*, 1903, **36**, 805.
Pfannstiel, Janecke, *Ber.*, 1942, **75**, 1096.

Phenylhydrazine-*m*-carboxylic Acid (*m-Hydrazinobenzoic acid*).

Pale yellow leaflets. M.p. 186° decomp. Spar. sol. EtOH, hot H_2O. Insol. Et_2O. Reacts strongly acid. Reduces Fehling's.

Roder, *Ann.*, 1886, **236**, 164.

Phenylhydrazine-*p*-carboxylic Acid (p-*Hydrazinobenzoic acid*).

Yellow needles or plates from H_2O. M.p. 114° (220–5° decomp.). Mod. sol. hot H_2O.

B,HCl: m.p. 252–3° (rapid heat).

Fischer, *Ann.*, 1882, **212**, 337.
Anchel, Schoenheimer, *J. Biol. Chem.*, 1936, **114**, 543.
Veibel, Hauge, *Bull. soc. chim.*, 1938, **5**, 1506.

Phenylhydrazine-β-sulphonic Acid

$C_6H_5{\cdot}NH{\cdot}NH{\cdot}SO_3H$

$C_6H_8O_3N_2S$ MW, 188

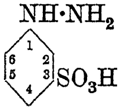
Not known in free state.

NH_4 salt: needles from EtOH. M.p. 208° decomp. Sol. H_2O. Mod. sol. EtOH. Insol. Et_2O, C_6H_6.

Na salt: leaflets from H_2O.

Paal, Kretschmer, *Ber.*, 1894, **27**, 1245.
Bucherer, Schmidt, *J. prakt. Chem.*, 1909, **79**, 402.

Phenylhydrazine-*m*-sulphonic Acid (*Hydrazinobenzene-*m*-sulphonic acid*)



$C_6H_8O_3N_2S$ MW, 188

Plates or needles + $2H_2O$ from H_2O. Sol. 40 parts H_2O at 22°. Sol. hot H_2O. Spar. sol. EtOH, Et_2O.

Limpricht, *Ber.*, 1888, **21**, 3409.

Phenylhydrazine-*p*-sulphonic Acid (*Hydrazinobenzene-*p*-sulphonic acid*).

Needles or leaflets from H_2O. Sol. 200 parts H_2O at 11°, 30 parts H_2O at 100°. Spar. sol. EtOH.

Thompson, *J. Soc. Dyers Colourists*, 1921, **37**, 7.

α-Phenylhydrazinoacetic Acid

$C_6H_5{\cdot}N(NH_2){\cdot}CH_2{\cdot}COOH$

$C_8H_{10}O_2N_2$ MW, 166

Leaflets from EtOH. M.p. 168° decomp. Spar. sol. EtOH. Insol. Et_2O, $CHCl_3$, C_6H_6. Sol. dil. HNO_3 with red col. Reduces warm Fehling's.

B,HCl: needles. M.p. 170° decomp.

Et ester: $C_{10}H_{14}O_2N_2$. MW, 194. Oil. B.p. 157–61°/7 mm. Sol. EtOH, Et_2O, C_6H_6, ligroin. Insol. H_2O. Reduces hot Fehling's.

Amide: $C_8H_{11}ON_3$. MW, 165. Needles. M.p. 150°. Sol. C_6H_6.

Anilide: needles from EtOH. M.p. 149°. Reduces Fehling's and $NH_3.AgNO_3$ in the cold.

Rupe, Heberlein, *Ann.*, 1898, **301**, 58.
Busch, *Ber.*, 1903, **36**, 3880.
Harries, *Ber.*, 1895, **28**, 1225.

β-Phenylhydrazinoacetic Acid

$C_6H_5{\cdot}NH{\cdot}NH{\cdot}CH_2{\cdot}COOH$

$C_8H_{10}O_2N_2$ MW, 166

Yellow leaflets or needles from EtOH. M.p. 153° (173° decomp.). Mod. sol. EtOH. Spar. sol. Me_2CO, AcOEt, C_6H_6.

B,HCl: leaflets. M.p. 165° decomp.

Et ester: oil. *Oxalate*: leaflets. M.p. 156°.

Phenyl ester: $C_{14}H_{14}O_2N_2$. MW, 242. Leaflets. M.p. 93–4°.

Anilide: m.p. 135°.

Rupe, Heberlein, *Ann.*, 1898, **301**, 58.
Busch, *Ber.*, 1903, **36**, 3880.
Busch, Meussdörfer, *J. prakt. Chem.*, 1907, **75**, 124.
Ghosh, Guha, *Chem. Zentr.*, 1934, I, 3050.

1-β-Phenylhydrazinobutyric Acid

$C_6H_5{\cdot}NH{\cdot}NH$ and $CH_3{\cdot}CH_2$ > $CH{\cdot}COOH$

$C_{10}H_{14}O_2N_2$ MW, 194

Needles from MeOH. Sublimes.

Amide: $C_{10}H_{15}ON_3$. MW, 193. Needles from Et_2O–pet. ether. M.p. 79°. Sol. EtOH, Et_2O. Less sol. C_6H_6.

Nitrile: $C_{10}H_{13}N_3$. MW, 175. Cryst. from Et_2O–pet. ether. M.p. 37°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O.

v. Miller, Plöchl, Sender, *Ber.*, 1892, **25**, 2037.

2-α-Phenylhydrazinobutyric Acid

$C_6H_5{\cdot}N(NH_2){\cdot}CH(CH_3){\cdot}CH_2{\cdot}COOH$

$C_{10}H_{14}O_2N_2$ MW, 194

Plates from EtOH. M.p. 111°. Sol. EtOH, $CHCl_3$. Spar. sol. H_2O, Et_2O.

Lederer, *J. prakt. Chem.*, 1891, **45**, 87.

2-β-Phenylhydrazinobutyric Acid

$C_6H_5{\cdot}NH{\cdot}NH{\cdot}CH(CH_3){\cdot}CH_2{\cdot}COOH$

$C_{10}H_{14}O_2N_2$ MW, 194

Needles from C_6H_6. M.p. 96–7°. Sol. EtOH, Et_2O. Insol. ligroin.

Prentice, *J. Chem. Soc.*, 1904, **85**, 1671.

Phenylhydrazinoformic Acid.

See Phenylhydrazine-carboxylic Acid.

1-α-Phenylhydrazinoisobutyric Acid

$C_6H_5{\cdot}N(NH_2){\cdot}C(CH_3)_2{\cdot}COOH$

$C_{10}H_{14}O_2N_2$ MW, 194

Amide: $C_{10}H_{15}ON_3$. MW, 193. Needles from C_6H_6. M.p. 118°. Sol. EtOH, C_6H_6, hot H_2O. Spar. sol. ligroin. *B,HCl*: needles from EtOH. M.p. 206°.

v. Walther, Hübner, *J. prakt. Chem.*, 1916, **93**, 132.

1-β-Phenylhydrazinoisobutyric Acid

$$C_6H_5{\cdot}NH{\cdot}NH{\cdot}C(CH_3)_2{\cdot}COOH$$

$C_{10}H_{14}O_2N_2$ MW, 194

Leaflets from EtOH. M.p. 165–6°.

Amide: leaflets from EtOH. M.p. 117°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O, ligroin. Reduces Fehling's on heating.

Nitrile: $C_{10}H_{13}N_3$. MW, 175. Needles from ligroin. M.p. 70°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Insol. H_2O. Reduces hot Fehling's.

Reissert, *Ber.*, 1884, **17**, 1461.
Eckstein, *Ber.*, 1892, **25**, 3323.

1-β-Phenylhydrazinopropionic Acid

$$C_6H_5{\cdot}NH{\cdot}NH{\cdot}CH(CH_3){\cdot}COOH$$

$C_9H_{12}O_2N_2$ MW, 180

Needles from MeOH. M.p. 174°.

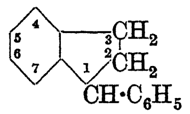
Et ester: $C_{11}H_{16}O_2N_2$. MW, 208. Liq. Very sol. dil. HCl. Reduces Fehling's in the cold.

Amide: $C_9H_{13}ON_3$. MW, 179. Cryst. from H_2O. M.p. 124°. Sol. EtOH, hot H_2O. Spar. sol. $CHCl_3$, C_6H_6, ligroin.

Nitrile: $C_9H_{11}N_3$. MW, 161. Cryst. from ligroin. M.p. 58°. Very sol. EtOH, Et_2O. Less sol. C_6H_6. Spar. sol. ligroin. Insol. H_2O. Reduces hot Fehling's. Fuming HCl ⟶ amide.

Reissert, *Ber.*, 1884, **17**, 1453; 1892, **25**, 2701.
v. Miller, Plöchl, Rohde, *Ber.*, 1892, **25**, 2060.

1-Phenylhydrindene



$C_{15}H_{14}$ MW, 194

B.p. 148–50°/13 mm.

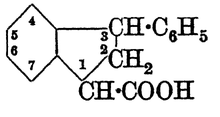
Mayer, Stieglitz, *Ber.*, 1921, **54**, 1399.

2-Phenylhydrindene.

Yellow oil. B.p. 162–3°/10 mm. D_4^{16} 1·0821. n_D^{15} 1·5955.

v. Braun, Manz, *Ber.*, 1929, **62**, 1062.

3-Phenyl-1-hydrindenic Acid (3-*Phenylhydrindene*-1-*carboxylic acid*)



$C_{16}H_{14}O_2$ MW, 238

Needles from AcOH. M.p. 143–144·5°. B.p. 227–9°/14 mm.

Me ester: $C_{17}H_{16}O_2$. MW, 252. Needles from EtOH–pet. ether. M.p. 77–8°. B.p. 204·5–205°/13 mm.

Et ester: $C_{18}H_{18}O_2$. MW, 266. Prisms from EtOH. M.p. 69–70°.

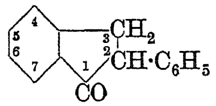
Blum-Bergmann, *Ann.*, 1930, **484**, 38; 1932, **492**, 277.

2-Phenyl-2-hydrindenic Acid (2-*Phenylhydrindene*-2-*carboxylic acid*).

Cryst. M.p. 194–5°.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 715.
Tilford, Campen, Shelton, *J. Am. Chem. Soc.*, 1947, **69**, 2902.

2-Phenyl-1-hydrindone (2-*Phenylindanone*)



$C_{15}H_{12}O$ MW, 208

Prisms from EtOH. M.p. 77–8°. B.p. 344° decomp. Very sol. $CHCl_3$, Me_2CO, C_6H_6. Sol. EtOH, Et_2O. Spar. sol. ligroin. Reduces warm $NH_3.AgNO_3$.

Semicarbazone: cryst. from MeOH. M.p. 211–12°.

Phenylhydrazone: cryst. from C_6H_6. M.p. 137–8°.

p-*Nitrophenylhydrazone*: yellow leaflets from Me_2CO, red cryst. powder from AcOH. M.p. 174°.

v. Miller, Rohde, *Ber.*, 1892, **25**, 2096.
Auwers, Auffenberg, *Ber.*, 1919, **52**, 107.
Plattner, Sandrin, Wyss, *Helv. Chim. Acta*, 1946, **29**, 1604.

3-Phenyl-1-hydrindone (3-*Phenylindanone*).

Prisms from MeOH. M.p. 78°. Very sol. EtOH, Et_2O, AcOH, C_6H_6.

Oxime: needles from EtOH.Aq. M.p. 141°.

Semicarbazone: m.p. 223–5°. Spar. sol. most solvents.

Phenylhydrazone: yellowish cryst. from EtOH. M.p. 130°.

p-*Nitrophenylhydrazone*: red cryst. from AcOH. M.p. 220–1°.

Auwers, Auffenberg, *Ber.*, 1919, **52**, 110.
Liebermann, Hartmann, *Ber.*, 1892, **25**, 2124.
Pfeiffer, de Waal, *Ann.*, 1935, **520**, 189.

4-Phenyl-1-hydrindone (4-*Phenylindanone*).

Thick yellow oil. B.p. 200–5°/11 mm.

Semicarbazone: cryst. M.p. 192°. Very sol. EtOH.

v. Braun, Manz, *Ann.*, 1929, **468**, 276.

Phenylhydrocinnamic Acid.

See 1 : 2-, and 2 : 2-Diphenylpropionic Acids.

Phenyl hydrogen sulphate

$$C_6H_5{\cdot}O{\cdot}SO_3H$$

$C_6H_6O_4S$ MW, 174

Very unstable.

Na salt: needles. Very hygroscopic. Decomp. easily.

K salt: leaflets from EtOH. Sol. 7 parts H_2O at 15°.

NH_4 salt: cryst. from dil. NH_3.Aq. $FeCl_3$ ⟶ reddish-violet col.

p-*Toluidine salt*: m.p. 145–6°.

Baumann, *Ber.*, 1878, **11**, 1907.
Czapek, *Monatsh.*, 1914, **35**, 639.
Hofmann, Biesalski, *Ber.*, 1912, **45**, 1396.
Feigenbaum, Neuberg, *J. Am. Chem. Soc.*, 1941, **63**, 3529.

3-Phenylhydroxyacetone (*Phenylacetylcarbinol, phenacetylcarbinol, hydroxymethyl benzyl ketone, phenylacetol,* 1-*hydroxy*-3-*phenylacetone, phenylpropanolone*)

$C_6H_5{\cdot}CH_2{\cdot}CO{\cdot}CH_2OH$

$C_9H_{10}O_2$ MW, 150

M.p. 48°. B.p. 144–5°/12–13 mm. Ag_2O ⟶ phenylacetic acid.

Me ether: $C_{10}H_{12}O_2$. MW, 164. B.p. 139–40°/27 mm. *Semicarbazone*: m.p. 127–8°.

Et ether: $C_{11}H_{14}O_2$. MW, 178. B.p. 116–17°/1 mm. *Semicarbazone*: m.p. 100°.

Phenyl ether: $C_{15}H_{14}O_2$. MW, 226. Needles from ligroin. M.p. 43–4°. *Semicarbazone*: needles from MeOH. M.p. 151–2°. *Phenylhydrazone*: yellow cryst. from MeOH. Sinters at 65°, m.p. 94–6°.

Benzyl ether: $C_{16}H_{16}O_2$. MW, 240. B.p. 235°/40 mm. *Semicarbazone*: m.p. about 105°.

Oxime: m.p. 118°.

Semicarbazone: m.p. 134°.

Acetyl: needles from EtOH. M.p. 131°.

p-*Nitrobenzoyl*: plates from EtOH.Aq. M.p. 120°.

Phenylurethane: m.p. 140°.

Darmon, *Compt. rend.*, 1933, **197**, 1328, 1649.

Phenyl hydroxybenzyl Ketone.

See Hydroxydeoxybenzoin.

β-Phenylhydroxylamine (N-*Phenylhydroxylamine*)

$C_6H_5{\cdot}NH{\cdot}OH$

C_6H_7ON MW, 109

Needles from H_2O, C_6H_6 or pet. ether. M.p. 81–2°. Sol. 50 parts cold, 10 parts hot H_2O. Very sol. EtOH, Et_2O, $CHCl_3$, CS_2, hot C_6H_6. Spar. sol. ligroin. Conc. H_2SO_4 ⟶ deep blue sol.

N-*Formyl*: plates or needles. M.p. 70–1°. Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O. Spar. sol. pet. ether.

N-*Acetyl*: needles from ligroin. M.p. 67–67·5°. Sol. EtOH, Et_2O, C_6H_6, hot H_2O. Spar. sol. pet. ether.

O:N-*Diacetyl*: prisms from C_6H_6–pet. ether. M.p. 43°. Sol. EtOH, Et_2O, Me_2CO, C_6H_6, boiling ligroin. Spar. sol. H_2O.

N-*Benzoyl*: needles from pet. ether. M.p. 123–4°. Spar. sol. Et_2O.

O:N-*Dibenzoyl*: cryst. from EtOH. M.p. 121°. Sol. hot EtOH, Et_2O, C_6H_6. Spar. sol. pet. ether.

N-*Carbethoxyl*: prisms from ligroin. M.p. 47·5°. B.p. 160–3°/12 mm. Sol. EtOH, Et_2O, Me_2CO, C_6H_6. Spar. sol. ligroin.

N-*Nitroso*: phenylisonitramine, phenylnitrosohydroxylamine. Needles from ligroin. M.p. 58–9°. Sol. usual solvents. Spar. sol. H_2O. $k = 5{\cdot}7 \times 10^{-6}$ at 0°. *NH_4 salt*: cupferron. Glistening white plates. Reagent for separation of Fe, Cu, titanium, vanadium, zirconium, uranium.

Wohl, *Ber.*, 1894, **27**, 1435.
Bamberger, Destraz, *Ber.*, 1902, **35**, 1884.
Ciamician, Silber, *Ber.*, 1905, **38**, 1183.
Kamm, *Organic Syntheses*, Collective Vol. I, 435.
Marvel, *ibid.*, 171.

Phenyl-hydroxynaphthylmethane.

See Benzylnaphthol.

Phenyl-hydroxyphenyl-acrylic Acid.

See Hydroxystilbene-α-carboxylic Acid.

Phenyl hydroxyphenyl Diketone.

See Hydroxybenzil.

Phenyl hydroxystyryl Ketone.

See 2-, 3-, and 4-Hydroxychalkones.

Phenyl ω-hydroxy-*p*-tolyl Ketone.

See 4-Hydroxymethyl-benzophenone.

Phenyliminazole.

See Phenylglyoxaline.

1-Phenyliminodiacetic Acid (*Phenyl*-ω-*carboxysarcosine, phenyl*-N-*carboxymethyl-glycine*)

$HOOC{\cdot}CH_2{\cdot}NH{\cdot}CH(C_6H_5){\cdot}COOH$

$C_{10}H_{11}O_4N$ MW, 209

Needles from H_2O. M.p. 220° decomp. Spar. sol. H_2O. Insol. EtOH, Et_2O.

B,HCl: leaflets. Decomp. at 220–2°.

Di-Me ester: $C_{12}H_{15}O_4N$. MW, 237. Thick oil. B.p. 220–1°/17 mm. D_4^{16} 1·1705, D_4^{25} 1·1622. n^{18} 1·5111.

Di-Et ester: $C_{14}H_{19}O_4N$. MW, 265. B.p. 195–6°/17 mm. Sol. EtOH, Et_2O. Insol. H_2O. D_4^{20} 1·1059. n^{20} 1·4976.

Diamide: $C_{10}H_{13}O_2N_3$. MW, 207. Cryst. M.p. 152–3°.

Stadnikow, *Ber.*, 1908, **41**, 4364; *Chem. Zentr.*, 1909, II, 1989.

N-Phenyliminodiacetic Acid (*Anil-diacetic acid, anilinodiacetic acid*)

$C_6H_5{\cdot}N(CH_2{\cdot}COOH)_2$

$C_{10}H_{11}O_4N$ MW, 209

Needles. M.p. 152–5° decomp. Sol. EtOH, hot H_2O. Rather spar. sol. Et_2O. $k = 2{\cdot}73 \times 10^{-4}$ at 25°. Alc. $FeCl_3$ ⟶ reddish-brown col. ⟶ brown ppt. on standing.

Di-Me ester: $C_{12}H_{15}O_4N$. MW, 237. B.p. 216–18°/25 mm., 210–11°/18 mm.
Mono-Et ester: $C_{12}H_{15}O_4N$. MW, 237. Cryst. from H_2O. M.p. 121–2°. B.p. about 230–40°/17 mm. *Anilide*: needles from Et_2O. M.p. 121–2°.
Di-Et ester: $C_{14}H_{19}O_4N$. MW, 265. Oil. B.p. 197–8°/17 mm. (200–6°/6 mm.).
Diamide: $C_{10}H_{13}O_2N_3$. MW, 207. Cryst. from H_2O. M.p. 238° (225°). Spar. sol. EtOH, Et_2O, $CHCl_3$.
Monoanilide: needles from EtOH.Aq. M.p. 215°.
Dianilide: needles from EtOH. M.p. 218°.

Vorländer, Mumme, *Ber.*, 1901, **34**, 1647.
de Mouilpied, *J. Chem. Soc.*, 1905, **87**, 439.
Johnson, Bengis, *J. Am. Chem. Soc.*, 1911, **33**, 749.

Phenyliminophosgene (*Phenylcarbylamine chloride*)

$C_6H_5{\cdot}N{:}CCl_2$

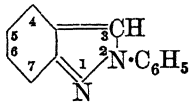
$C_7H_5NCl_2$ MW, 174

B.p. 209–11°, 104–6°/30 mm. D_4^{15} 1·285. Vapour is toxic and lachrymatory.

Bly, Perkins, Lewis, *J. Am. Chem. Soc.*, 1922, **44**, 2896.
Dyson, Harrington, *J. Chem. Soc.*, 1940, 191.

Phenyliminopropionic Acid.
See under Pyruvic Acid.

2-Phenylindazole



$C_{13}H_{10}N_2$ MW, 194

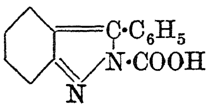
Needles from EtOH. M.p. 83–4°. B.p. 344–5°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Spar. sol. ligroin. Insol. H_2O. Sol. conc. min. acids. CrO_3 in AcOH, or $KMnO_4$ ⟶ azobenzene-2-carboxylic acid.
B_2,H_2PtCl_6: m.p. 187–8° decomp.
Picrate: yellow needles. M.p. 93–4°.

Reissert, Lemmer, *Ber.*, 1926, **59**, 356.
Paal, *Ber.*, 1891, **24**, 961.
Busch, *Ber.*, 1894, **27**, 2899.
Freundler, *Bull soc. chim.*, 1903, **29**, 745; 1904, **31**, 868.

3-Phenylindazole.
Exists in two forms.
(i) Needles from ligroin. M.p. 107–8°. Heated above m.p. ⟶ (ii).
(ii) Prisms from ligroin. M.p. 115–16°. Heated to higher temps. again ⟶ (i).
1(?)N-*Acetyl*: needles from ligroin. M.p. 69–79°.

Auwers, Schaum, *Ber.*, 1929, **62**, 1672.

3-Phenylindazole-2-carboxylic Acid

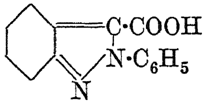


$C_{14}H_{10}O_2N_2$ MW, 238

Me ester: $C_{15}H_{12}O_2N_2$. MW, 252. Needles from MeOH. M.p. 112–13°. Sol. EtOH, AcOH. Prac. insol. C_6H_6, pet. ether.
Et ester: $C_{16}H_{14}O_2N_2$. MW, 266. Needles. M.p. 83–4°.

Auwers, Hüttenes, *Ber.*, 1922, **55**, 1133.

2-Phenylindazole-3-carboxylic Acid

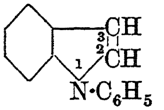


$C_{14}H_{10}O_2N_2$ MW, 238

M.p. 200° decomp. Heat ⟶ 2-phenylindazole.
Me ester: needles from EtOH. M.p. 169–70°.
Amide: $C_{14}H_{11}ON_3$. MW, 237. M.p. 247–8° (243–4°).
Nitrile: $C_{14}H_9N_3$. MW, 219. M.p. 105° (106–7°).

Reissert, Lemmer, *Ber.*, 1926, **59**, 355.
Heller, Spielmeyer, *Ber.*, 1925, **58**, 836.

1-Phenylindole (N-*Phenylindole*)

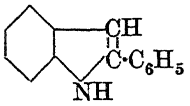


$C_{14}H_{11}N$ MW, 193

Yellowish oil. B.p. 326–7°/757 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Volatile in steam. With pine-splinter + HCl ⟶ bluish-violet col.

Fischer, Hess, *Ber.*, 1884, **17**, 568.
Pfülf, *Ann.*, 1887, **239**, 221.

2-Phenylindole (*α-Phenylindole*)



$C_{14}H_{11}N$ MW, 193

Leaflets from EtOH or ligroin. M.p. 189° (187°). B.p. above 360°, about 250°/10 mm. Odour resembles indole. Gradually turns green in air. Sublimes. Spar. volatile in steam. Sol. Et_2O, $CHCl_3$, AcOH, C_6H_6, hot CS_2. Spar. sol. hot H_2O. Insol. dil. min. acids. CrO_3 in boiling AcOH ⟶ benzoic acid.
N-*Me*: $C_{15}H_{13}N$. MW, 207. Cryst. from EtOH, C_6H_6, or ligroin. M.p. 101°. Spar. volatile in steam. Mod. sol. cold conc. HCl, re-pptd. unchanged on dilution.
N-*Et*: $C_{16}H_{15}N$. MW, 221. Needles from EtOH. M.p. 86°.

Picrate: red prisms from EtOH. M.p. 127°.

I.C.I., B.P. 330,332, (*Chem. Abstracts*, 1930, **24**, 5770).
I.G., D.R.P., 574,840, (*Chem. Abstracts*, 1933, **27**, 4541).
Campbell, *J. Chem. Soc.*, 1935, 1210.
Wolff, *Ann.*, 1912, **394**, 107.
Madelung, *Ber.*, 1912, **45**, 1131.
Fischer, Hütz, *Ber.*, 1895, **28**, 587.
Shriner, Ashley, Welch, *Organic Syntheses*, 1942, XXII, 98.
Ruggli, Hegediio, *Helv. Chim. Acta*, 1939, **22**, 405.

3-Phenylindole (β-*Phenylindole*).

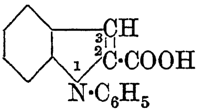
Leaflets from ligroin. M.p. 88–9°. Sol. EtOH, Et_2O, C_6H_6. Mod. sol. hot ligroin. Insol. H_2O. $ZnCl_2$ at 170° ⟶ 2-phenylindole.

N-*Me*: cryst. from pet. ether. M.p. 64–5°. Sol. EtOH, Et_2O, C_6H_6. $ZnCl_2$ at 210° ⟶ *N*-methyl-2-phenylindole. *Picrate*: brown needles from C_6H_6–ligroin. M.p. 90°.

N-*Nitroso*: yellow needles from ligroin. M.p. 60–1°. Sol. Et_2O, $CHCl_3$, Me_2CO, C_6H_6. *Picrate*: red needles from C_6H_6–ligroin. M.p. 107°.

Fischer, Schmidt, *Ber.*, 1888, **21**, 1073, 1811.
Henle, *Ber.*, 1905, **38**, 1365.
Ince, *Ann.*, 1889, **253**, 38.

1-Phenylindole-2-carboxylic Acid



$C_{15}H_{11}O_2N$ MW, 237

Needles from EtOH.Aq. M.p. 176°. Sol. EtOH, Et_2O. Very spar. sol. boiling H_2O. At 200–10° ⟶ 1-phenylindole.

Fischer, Hess, *Ber.*, 1884, **17**, 567.
Pfülf, *Ann.*, 1887, **239**, 221.

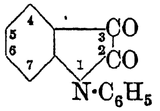
3-Phenylindole-2-carboxylic Acid.

Needles from C_6H_6. M.p. 186°.

Et ester: $C_{17}H_{15}O_2N$. MW, 265. Cryst. from EtOH. M.p. 137–8°.

Manske, Perkin, Robinson, *J. Chem. Soc.*, 1927, 6.

***N*-Phenylisatin**



$C_{14}H_9O_2N$ MW, 223

Yellowish-red leaflets from EtOH. M.p. 138°. Mod. sol. EtOH, Et_2O, C_6H_6.

3-*Hydrazone*: yellow needles from EtOH. M.p. 192°.

Stollé, *Ber.*, 1913, **46**, 3915; D.R.P., 281,046, (*Chem. Zentr.*, 1915, I, 71).
Pfülf, *Ann.*, 1887, **239**, 222.

1-Phenylisoamyl Alcohol.

See Isobutylphenylcarbinol.

2-Phenylisoamyl Alcohol (2-*Methyl*-3-*phenylbutanol*-4, 3-*methyl*-2-*phenyl*-n-*butyl alcohol*)

$$(CH_3)_2CH{\cdot}\underset{}{\overset{C_6H_5}{\overset{|}{C}}}H{\cdot}CH_2OH$$

$C_{11}H_{16}O$ MW, 164

Thick oil. B.p. 130°/15 mm.

Acetyl: liq. B.p. 134°/15 mm.

Ramart-Lucas, Amagat, *Compt. rend.*, 1927, **184**, 30.
Blaise, Courtot, *Bull. soc. chim.*, 1906, **35**, 595.

***N*-Phenylisoamylamine.**

See Isoamylaniline.

1-Phenylisobutane.

See Isobutylbenzene.

2-Phenylisobutane.

See *tert.*-Butylbenzene.

2-Phenylisobutyl Alcohol

$$(CH_3)_2\overset{C_6H_5}{\overset{|}{C}}{\cdot}CH_2OH$$

$C_{10}H_{14}O$ MW, 150

B.p. 131°/30 mm., 122–3°/20 mm. n_D^{20} 1·5261.

Phenylurethane: m.p. 59–60°.

α-*Naphthylurethane*: m.p. 91·5–92·5°.

Haller, Bauer, *Ann. chim.*, 1918, **9**, 10.
Whitmore, Weisgerber, Shabica, *J. Am. Chem. Soc.*, 1943, **65**, 1469.

Phenylisobutylamine.

See Aminoisobutylbenzene *and* *N*-Isobutylaniline.

1-Phenylisobutylene (ββ-*Dimethylstyrene*)

$$C_6H_5{\cdot}CH{:}C{<}^{CH_3}_{CH_3}$$

$C_{10}H_{12}$ MW, 132

M.p. − 52°. B.p. 183–5°, 76–7°/11 mm. D_4^{20} 0·8980. n_D^{20} 1·5273.

Nitrosite: needles from C_6H_6. M.p. 122° decomp. (154° decomp.).

Lévy, Tabart, *Bull. soc. chim.*, 1931, **49**, 1776.

1-Phenylisobutyraldehyde (*Dimethylphenylacetaldehyde*)

$$C_6H_5{\cdot}\overset{CH_3}{\underset{CH_3}{\overset{|}{\underset{|}{C}}}}{\cdot}CHO$$

$C_{10}H_{12}O$ MW, 148

B.p. 215–18°, 105–10°/14 mm. D^0 0·9912. Does not form bisulphite comp.

Semicarbazone: m.p. 172°.

Tiffeneau, Dorlencourt, *Ann. chim. phys.*, 1907, **10**, 366; 1909, **16**, 248.

2-Phenylisobutyraldehyde.
See α-Methylhydrocinnamaldehyde.

1-Phenylisobutyric Acid (*1-Methylhydratropic acid, dimethylphenylacetic acid*)

$$C_6H_5\cdot C(CH_3)_2\cdot COOH$$

$C_{10}H_{12}O_2$ MW, 164

Prisms from EtOH. M.p. 80–1°. B.p. 150–5°/10 mm. Conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Me ester: $C_{11}H_{14}O_2$. MW, 178. B.p. 225°.
Et ester: $C_{12}H_{16}O_2$. MW, 192. B.p. 235–6°.
Propyl ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 250°.
Isobutyl ester: $C_{14}H_{20}O_2$. MW, 220. B.p. 260–1°.
Chloride: $C_{10}H_{11}OCl$. MW, 182·5. B.p. 109°/13 mm.
Amide: $C_{10}H_{13}ON$. MW, 163. Cryst. M.p. 160–1°. B.p. 200–5°/60 mm. Spar. sol. Et_2O. Insol. H_2O.
Nitrile: $C_{10}H_{11}N$. MW, 145. B.p. 232°, 114–16°/20 mm. D^{21} 0·966. n_D 1·50665.

Wallach, *Chem. Zentr.*, 1899, II, 1047.
Haller, Bauer, *Compt. rend.*, 1912, **155**, 1582.

2-Phenylisobutyric Acid.
See α-Methylhydrocinnamic Acid.

1-Phenylisocaproic Acid (*3-Methyl-1-phenylvaleric acid, isobutylphenylacetic acid*)

$$(CH_3)_2CH\cdot CH_2\cdot CH(C_6H_5)\cdot COOH$$

$C_{12}H_{16}O_2$ MW, 192

Prisms from pet. ether. M.p. 78–9°.
Nitrile: $C_{12}H_{15}N$. MW, 173. B.p. 263–6°, 136–8°/15 mm. D^{16} 0·942. Sol. EtOH, Et_2O, C_6H_6, AcOH. Insol. H_2O. Volatile in steam.

Bodroux, Taboury, *Bull. soc. chim.*, 1910, **7**, 668.

3-Phenylisocaproic Acid (*3-Methyl-3-phenylvaleric acid*)

$$(CH_3)_2C(C_6H_5)\cdot CH_2\cdot CH_2\cdot COOH$$

$C_{12}H_{16}O_2$ MW, 192

B.p. 130–140°/1 mm.
Anilide: m.p. 116–17°.

Späth, Kainrath, *Ber.*, 1938, **71**, 1662.

4-Phenylisocaproic Acid (*3-Methyl-4-phenylvaleric acid, 3-benzylvaleric acid*)

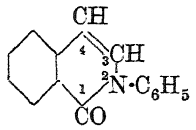
$$C_6H_5\cdot CH_2(CH_3)>CH\cdot CH_2\cdot CH_2\cdot COOH$$

$C_{12}H_{16}O_2$ MW, 192

B.p. 130–50°/1 mm.
Anilide: m.p. 74–6°.

Späth, Kainrath, *Ber.*, 1938, **71**, 1662.

***N*-Phenylisocarbostyril** (*2-Phenylisoquinolone*)

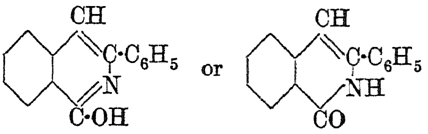


$C_{15}H_{11}ON$ MW, 221

Needles from EtOH.Aq. M.p. 117·5°. Very sol. EtOH. Spar. sol. Et_2O, C_6H_6. Sublimes.

Bamberger, Frew, *Ber.*, 1894, **27**, 203.
Dieckmann, Meiser, *Ber.*, 1908, **41**, 3268.

3-Phenylisocarbostyril (*1-Hydroxy-3-phenylisoquinoline, 3-phenylisoquinolone*)



$C_{15}H_{11}ON$ MW, 221

Needles or prisms. M.p. 197°. Sol. hot EtOH, conc. HCl. Dist. with Zn dust in H ⟶ 3-phenylisoquinoline.
Et ether: $C_{17}H_{15}ON$. MW, 249. Needles from EtOH. M.p. 45–6°.

Gabriel, *Ber.*, 1886, **19**, 835.
Wölbling, *Ber.*, 1905, **38**, 3848.

2-Phenylisocrotonic Acid.
Cis form of β-Methylcinnamic Acid. *See* β-Methylcinnamic Acid.

Phenyl isocyanate (*Phenylcarbimide, carbanil*)

$$C_6H_5\cdot N{:}CO$$

C_7H_5ON MW, 119

Pungent lachrymatory liq. B.p. 162–3°/751 mm., 55°/13 mm. $D_4^{19\cdot6}$ 1·0956, $D_4^{25\cdot9}$ 1·08870. $n_D^{19\cdot6}$ 1·53684. Used for characterisation of alcohols and as a dehydrating agent.

Hardy, *J. Chem. Soc.*, 1934, 2011.
Zimmer, D.R.P., 133,760, (*Chem. Zentr.*, 1902, II, 553).

Phenyl isocyanide (*Phenylcarbylamine, benzoisonitrile*)

$$C_6H_5\cdot NC$$

C_7H_5N MW, 103

Liq. with penetrating odour. B.p. 165–6° part. polymerisation, 80–1° (78°)/40 mm. D^{15} 0·977. Very unstable. Rapidly turns green and then blue ⟶ slowly to a brown resin. Na + amyl alcohol ⟶ methylaniline. Heated with EtONa in sealed tube ⟶ N : N′-diphenylformamidine.

Biddle, Goldberg, *Ann.*, 1900, **310**, 7.
Malatesta, *Gazz. chim. ital.*, 1947, **77**, 238.

2-Phenylisohexyl Alcohol (*2-Methyl-4-phenylpentanol-5*)

$$\begin{array}{c} C_6H_5 \\ | \\ (CH_3)_2CH{\cdot}CH_2{\cdot}CH{\cdot}CH_2OH \end{array}$$

$C_{12}H_{18}O$ MW, 178

B.p. 138–9°/14 mm.

Phenylurethane: cryst. from Et_2O–ligroin. M.p. 78°.

Ramart, Amagat, *Ann. chim.*, 1927, **8**, 288.

Phenylisonitramine.
See under Phenylhydroxylamine.

Phenylisonitromethane.
See under Phenylnitromethane.

2-Phenylisopentane.
See tert.-Amylbenzene.

3-Phenylisopentane (*2-Methyl-3-phenylbutane*)

$$\begin{array}{c} C_6H_5 \\ | \\ CH_3{\cdot}CH{\cdot}CH(CH_3)_2 \end{array}$$

$C_{11}H_{16}$ MW, 148

B.p. 188–9°/753 mm. D_4^{16} 0·8672. n_D^{16} 1·4972.

Konowalow, Jegorow, *Chem. Zentr.*, 1899, I, 776.
Klages, *Ber.*, 1903, **36**, 3691.

4-Phenylisopentane.
See Isoamylbenzene.

1-Phenyl-2-isopentene.
See 3-Methyl-1-phenyl-2-butylene.

Phenylisophthalic Acid.
See Diphenyl-dicarboxylic Acid.

Phenylisopropyl Alcohol.
See Dimethylphenylcarbinol *and* Methylbenzylcarbinol.

***N*-Phenylisopropylamine.**
See N-Isopropylaniline.

1-Phenylisopropylamine (*α-Aminocumene, 2-amino-2-phenylpropane, α-aminoisopropylbenzene*)

$$(CH_3)_2C(NH_2){\cdot}C_6H_5$$

$C_9H_{13}N$ MW, 135

B.p. 196–7°/762 mm. D_0^{20} 0·9424. n_D^{25} 1·5181.

B,HCl: m.p. 235·5°. Sol. EtOH. Very spar. sol. Et_2O, C_6H_6. Insol. pet. ether.

B,HNO₂: m.p. 98–9° decomp.

N-*Benzoyl*: needles from EtOH. M.p. 159°. Very spar. sol. Et_2O.

Oxalate: needles from EtOH. M.p. 131°.

Konowalow, *Chem. Zentr.*, 1894, II, 33.
Brander, *Rec. trav. chim.*, 1918, **37**, 68.

2-Phenylisopropylamine (*2-Amino-1-phenylpropane, 1-benzylethylamine, β-aminopropylbenzene, amphetamine, benzedrine, elastonan*)

$$C_6H_5{\cdot}CH_2{\cdot}CH(NH_2){\cdot}CH_3$$

$C_9H_{13}N$ MW, 135

d-.

B.p. 102°/16 mm., 80°/10 mm. D_4^{15} 0·949. n_D^{20} 1·4704. $[\alpha]_D^{15}$ + 35·6°.

B,HCl: m.p. 156°. $[\alpha]_D^{15}$ + 24·8° in H_2O.

N-*Benzoyl*: cryst. from EtOH. M.p. 159–60°. $[\alpha]_D^{15}$ −14·8° in C_6H_6.

dl-.

B.p. 205° (203°).

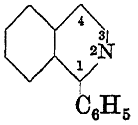
B,HCl: m.p. 145–7°. Hygroscopic.

N-*Acetyl*: needles from EtOH.Aq. M.p. 64° initially, 93° on standing.

Picrate: m.p. 143°.

Hey, *J. Chem. Soc.*, 1930, 18.
Jones, Wallis, *J. Am. Chem. Soc.*, 1926, **48**, 180.
Leithe, *Ber.*, 1932, **65**, 664.
Jaeger, van Dijk, *Chem. Abstracts*, 1943, **37**, 621.
Haskelberg, *J. Am. Chem. Soc.*, 1948, **70**, 2811.

1-Phenylisoquinoline



$C_{15}H_{11}N$ MW, 205

Needles from EtOH.Aq. M.p. 95–6°. B.p. 298°/729 mm., 120–40°/1 mm. Spar. volatile in steam.

B,HCl: needles from EtOH–Et_2O. M.p. 235–6°.

B₂,H₂PtCl₆: yellowish-red prisms from EtOH. M.p. 242–3° decomp.

Picrate: yellow needles from EtOH. M.p. 165°.

Pictet, Gams, *Ber.*, 1910, **43**, 2388.
Späth, Berger, Kuntara, *Ber.*, 1930, **63**, 139.
Rosenmund, Nothnagel, Riesenfeldt, *Ber.*, 1927, **60**, 395.
Davies, Kefford, Osborne, *J. Chem. Soc.*, 1939, 360.

3-Phenylisoquinoline.

Leaflets from EtOH. M.p. 103–5°. Spar. volatile in steam.

B,HAuCl₄,H₂O: m.p. 179–80°.

Gabriel, Neumann, *Ber.*, 1892, **25**, 3573.

4-Phenylisoquinoline.

M.p. 80°.

Picrate: m.p. 209°.

Krabbe, D.R.P. 652,041, (*Chem. Abstracts*, 1938, **32**, 1715).

Phenylisoquinolone.
See Phenylisocarbostyril.

2-Phenylisoserine.
See 1-Hydroxy-2-amino-2-phenylpropionic Acid.

1-Phenylisosuccinic Acid.
See Methylphenylmalonic Acid.

2-Phenylisosuccinic Acid.
See Benzylmalonic Acid.

Phenyl isothiocyanate

$C_6H_5{\cdot}N{:}CS$

C_7H_5NS MW, 135

Colourless liq. F.P. − 21°. B.p. 221°, 131·8°/63 mm., 120–1°/35 mm., 95°/12 mm. D_4^4 1·1477, D_4^{25} 1·1288, D_4^{35} 1·1202, D_4^{50} 1·1061. $n_D^{23\cdot4}$ 1·64918. Heat of comb. C_v 1019·0 Cal., C_p 1020·3 Cal.

Dains, Brewster, Olander, *Organic Syntheses*, Collective Vol. I, 437.

2-Phenylisovaleraldehyde.

See β : β-Dimethylhydrocinnamaldehyde.

1-Phenylisovaleric Acid (*Isopropylphenylacetic acid*)

$$(CH_3)_2CH{\cdot}\overset{C_6H_5}{\overset{|}{C}}H{\cdot}COOH$$

$C_{11}H_{14}O_2$ MW, 178

Prisms from ligroin. M.p. 61–2°. B.p. 159–60°/14 mm.

Amide: $C_{11}H_{15}ON$. MW, 177. Needles from EtOH.Aq. M.p. 111–12° (68°).

Nitrile: $C_{11}H_{13}N$. MW, 159. B.p. 245–9°/765 mm. $D^{15\cdot5}$ 0·967. Sol. EtOH, C_6H_6. Insol. H_2O. Volatile in steam.

Anilide: m.p. 132–3°.

Bodroux, Taboury, *Bull. soc. chim.*, 1910, **7**, 669.
Haller, Bauer, *Compt. rend.*, 1909, **149**, 9.
Hoffmann, *J. Am. Chem. Soc.*, 1929, **51**, 2546.

2-Phenylisovaleric Acid.

See β : β-Dimethylhydrocinnamic Acid.

3-Phenylisovaleric Acid (2-*Benzylbutyric acid*)

$$C_6H_5{\cdot}CH_2{\cdot}\overset{CH_3}{\overset{|}{C}}H{\cdot}CH_2{\cdot}COOH$$

$C_{11}H_{14}O_2$ MW, 178

Oil. B.p. 172°/13 mm. (161°/13 mm.). Sol. EtOH, Et_2O. Insol. H_2O.

Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 144–6°/15 mm.

Chloride: $C_{11}H_{13}OCl$. MW, 196·5. B.p. 133–5°/14 mm., 123°/12 mm.

Amide: m.p. 83°.

Nitrile: $C_{11}H_{13}N$. MW, 159. B.p. 121°/13 mm.

Anilide: needles from EtOH. M.p. 101°.

Anschütz, Motschmann, *Ann.*, 1915, **407**, 88.
v. Braun, Stuckenschmidt, *Ber.*, 1923, **56**, 1728.
Weygand, Schröder, *Ber.*, 1941, **74**, 1844.

Phenylitaconic Acid (*Benzylidenesuccinic acid, γ-phenylitaconic acid*)

$$\alpha\ HOOC{\cdot}CH_2{\cdot}\overset{C_6H_5{\cdot}CH}{\overset{||}{C}}{\cdot}COOH\ \beta$$

$C_{11}H_{10}O_4$ MW, 206

Plates from Et_2O. M.p. 192° (rapid heat.) with loss of H_2O. Sol. 24 parts boiling H_2O. Spar. sol. Et_2O, C_6H_6, cold H_2O. Very spar. sol. $CHCl_3$, CS_2. $k = 1{\cdot}37 \times 10^{-4}$ at 25°. Heat at 180–5° in vacuo ⟶ phenylitaconic anhydride + a little phenylcitraconic anhydride. $KMnO_4$ ⟶ benzaldehyde, malonic and oxalic acids.

Di-Me ester: $C_{13}H_{14}O_4$. MW, 234. Thick oil. B.p. 186°/19 mm. Misc. in all proportions with EtOH, Et_2O, Me_2CO, C_6H_6. Spar. sol. ligroin. Insol. H_2O. Volatile in steam.

α-Et ester: $C_{13}H_{14}O_4$. MW, 234. Needles from H_2O or pet. ether. M.p. 76–9°.

β-Et ester: needles or plates from CS_2. M.p. 72°. Sol. EtOH, Et_2O. Spar. sol. pet. ether, CS_2.

Di-Et ester: $C_{15}H_{18}O_4$. MW, 262. Oil. B.p. 315°, 195°/20 mm. Polymerises slowly in the dark, more rapidly in light. n_α^{20} 1·5243.

Anhydride: $C_{11}H_8O_3$. MW, 188. Leaflets from $CHCl_3$. M.p. 164–6° ⟶ phenylcitraconic anhydride with slight decomp. Sol. 9 parts boiling C_6H_6, 200 parts boiling Et_2O. Very spar. sol. pet. ether, CS_2.

Fittig, Brooke, *Ann.*, 1899, **305**, 21.
Stobbe, *Ber.*, 1908, **41**, 4353.
Hecht, *Monatsh.*, 1903, **24**, 367.

Phenyl-J Acid.

See *N*-Phenyl-2-amino-5-naphthol-7-sulphonic Acid.

1-Phenyl-lactic Acid.

See Atrolactinic Acid.

2-Phenyl-lactic Acid.

See α-Hydroxyhydrocinnamic Acid.

2-Phenyl-lepidine.

See Flavoline.

1-Phenyl-levulinic Acid (1-*Phenyl-2-acetylpropionic acid*)

$$CH_3{\cdot}CO{\cdot}CH_2{\cdot}\overset{C_6H_5}{\overset{|}{C}}H{\cdot}COOH$$

$C_{11}H_{12}O_3$ MW, 192

Leaflets from H_2O. M.p. 127°. Sol. EtOH, Et_2O.

Me ester: $C_{12}H_{14}O_3$. MW, 206. M.p. 71°.

Et ester: $C_{13}H_{16}O_3$. MW, 220. M.p. 41–2°. B.p. 167°/17 mm.

Amide: $C_{11}H_{13}O_2N$. MW, 191. Yellow prisms from AcOH.Aq. M.p. 236° decomp.

Semicarbazone: m.p. 215° (slow heat.).

Phenylhydrazone: needles from EtOH. M.p. 140°.

Weltner, *Ber.*, 1885, **18**, 790.
Ruhemann, *J. Chem. Soc.*, 1904, **85**, 1455.
Huan, *Bull. soc. chim.*, 1938, **5**, 1341.

2-Phenyl-levulinic Acid (2-*Phenyl-2-acetylpropionic acid*)

$$CH_3{\cdot}CO{\cdot}\overset{C_6H_5}{\overset{|}{C}}H{\cdot}CH_2{\cdot}COOH$$

$C_{11}H_{12}O_3$ MW, 192

M.p. 98°.
Semicarbazone: m.p. 224°.

Mukherji, *Chem. Abstracts*, 1948, **42**, 6323.

4-Phenyl-levulinic Acid (*2-Phenylacetyl-propionic acid*)

$C_6H_5 \cdot CH_2 \cdot CO \cdot CH_2 \cdot CH_2 \cdot COOH$

$C_{11}H_{12}O_3$ MW, 192

Needles from $CHCl_3$–ligroin. M.p. 55–6°. Sol. Et_2O, CS_2, C_6H_6. Mod. sol. H_2O. Spar. sol. ligroin.

Semicarbazone: needles from 50% EtOH. M.p. 182–3° decomp.

Stobbe, Russwurm, Schulz, *Ann.*, 1899, **308**, 179.

Phenylmaleic Acid (*1-Phenylethylene-1 : 2-dicarboxylic acid*)

$C_6H_5 \cdot C \cdot COOH$
$\quad\; HC \cdot COOH$

$C_{10}H_8O_4$ MW, 192

Prisms from Et_2O. Mod. sol. H_2O. Very unstable.

Anhydride: needles from CS_2. M.p. 119–119·5°. Sol. Et_2O, $CHCl_3$. Spar. sol. CS_2, ligroin. Cold $H_2O \longrightarrow$ acid.

Alexander, *Ann.*, 1890, **258**, 77.

1-Phenylmalic Acid

$C_6H_5 \cdot C(OH) \cdot COOH$
$\quad\; CH_2 \cdot COOH$

$C_{10}H_{10}O_5$ MW, 210

Prisms from $CHCl_3$. M.p. 187–8°. Sol. 75 parts H_2O at 15°. Sol. Et_2O.

Alexander, *Ann.*, 1890, **258**, 76.

2-Phenylmalic Acid

$HO \cdot CH \cdot COOH\ \alpha$
$C_6H_5 \cdot CH \cdot COOH\ \beta$

$C_{10}H_{10}O_5$ MW, 210

Cryst. from Et_2O. M.p. 160°. Sol. 3 parts H_2O at 15°. Insol. $CHCl_3$, C_6H_6, ligroin.

β-Et ester: $C_{12}H_{14}O_5$. MW, 238. Oil. *α-Nitrile*: $C_{12}H_{13}O_3N$. MW, 219. Prisms from hot EtOH. M.p. 127–8°. Sol. $CHCl_3$. Spar. sol. Et_2O, ligroin.

Alexander, *Ann.*, 1890, **258**, 80.
Börner, *Chem. Zentr.*, 1900, I, 123.

Phenylmalonaldehydic Acid.
See Phenylformylacetic Acid.

Phenylmalonic Acid

$C_6H_5 \cdot CH{<}^{COOH}_{COOH}$

$C_9H_8O_4$ MW, 180

Prisms from H_2O. M.p. 152–3° decomp. Sol. H_2O, EtOH, Et_2O, Less sol. $CHCl_3$, CS_2, C_6H_6, ligroin. Above m.p. $\longrightarrow$ phenylacetic acid.

Me ester: $C_{10}H_{10}O_4$. MW, 194. Cryst. from MeOH. M.p. 95°. *Anilide*: m.p. 109°.

Di-Me ester: $C_{11}H_{12}O_4$. MW, 208. Cryst. from ligroin. M.p. 51°. B.p. 147–9°/10 mm., 120–30°/3 mm.

Et ester: $C_{11}H_{12}O_4$. MW, 208. *Amide*: $C_{11}H_{13}O_3N$. MW, 207. Needles from C_6H_6. M.p. 152°. Sol. EtOH. Insol. H_2O.

Di-Et ester: $C_{13}H_{16}O_4$. MW, 236. M.p. 16–17°. B.p. 171°/21 mm., 158–9°/10 mm., 127–30°/2 mm.

Dichloride: $C_9H_6O_2Cl_2$. MW, 217. B.p. 122°/15 mm.

Diamide: $C_9H_{10}O_2N_2$. MW, 178. Cryst. from EtOH. M.p. 233°. Sol. EtOH. Insol. H_2O.

Nitrile: *see* Phenylcyanoacetic Acid.

Dinitrile: $C_9H_6N_2$. MW, 142. Cryst. from EtOH.Aq. M.p. 68–9°. B.p. 152–3°/21 mm. Sol. EtOH, Et_2O. Spar. sol. H_2O, ligroin.

Nelson, Cretcher, *J. Am. Chem. Soc.*, 1928, **50**, 2758.
Hessler, *Am. Chem. J.*, 1905, **32**, 123.
Wislicenus, *Ber.*, 1894, **27**, 1093.
Staudinger, Hirzel, *Ber.*, 1917, **50**, 1030.
Rising, Stieglitz, *J. Am. Chem. Soc.*, 1918, **40**, 728.
Dox, Yoder, *J. Am. Chem. Soc.*, 1922, **44**, 1564.
Ivanoff, Spassoff, *Bull. soc. chim.*, 1931, **49**, 19.
Blum-Bergmann, *Ber.*, 1932, **65**, 115.
Wallingford, Homeyer, Jones, *J. Am. Chem. Soc.*, 1941, **63**, 2056.
Walker, Levine, Kibler, Hauser, *J. Am. Chem. Soc.*, 1946, **68**, 672.

Phenyl Mercaptan.
See Thiophenol.

Phenylmercaptobenzoic Acid.
See Diphenyl sulphide carboxylic Acid.

Phenylmesaconic Acid (*Benzylfumaric acid*)

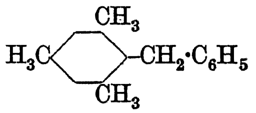
$C_6H_5 \cdot CH_2 \cdot C \cdot COOH$
$\quad HOOC \cdot CH$

$C_{11}H_{10}O_4$ MW, 206

Needles from H_2O. M.p. 212°. Sol. Et_2O, boiling C_6H_6. Sol. 35 parts boiling H_2O. Spar. sol. cold H_2O. Insol. $CHCl_3$, CS_2, ligroin. Heat with 10% NaOH $\longrightarrow$ phenylitaconic acid + phenylaticonic acid.

Fittig, Brooke, *Ann.*, 1899, **305**, 31.

Phenylmesitylmethane (*Benzylmesitylene, 1 : 3 : 5-trimethyldiphenylmethane*)



$C_{16}H_{18}$ MW, 210

Needles. M.p. 37°. B.p. 301–3°, 183°/11 mm. CrO_3 ⟶ benzoylmesitylene. HI + P ⟶ toluene + mesitylene.

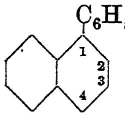
Louise, *Ann. chim. phys.*, 1885, **6**, 177.
Klages, Allendorff, *Ber.*, 1898, **31**, 1001.

Phenyl *p*-methoxyphenacyl Ether.
See under p-Hydroxyphenacyl Alcohol.

Phenyl methoxystyryl Ketone.
See Anisylideneacetophenone *and under* 2-, and 3-Hydroxychalkones.

Phenyl methylstyryl Ketone.
See Dypnone *and* 2′-, 3′-, and 4′-Methyl chalkones.

1-Phenylnaphthalene



$C_{16}H_{12}$ MW, 204

Cryst. M.p. about 45°. B.p. 334°/760 mm., 186–8°/10 mm. Sol. EtOH, Et_2O, AcOH, C_6H_6. Shows faint blue fluor.

Chattaway, *J. Chem. Soc.*, 1893, **63**, 1185.
Möhlau, Berger, *Ber.*, 1893, **26**, 1198.
Veselý, Štursa, *Chem. Zentr.*, 1936, I, 1221.
Weiss, *Organic Syntheses*, 1944, XXIV, 84.

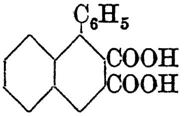
2-Phenylnaphthalene.
Leaflets from EtOH. M.p. 103–4°. B.p. 345–6°, 185–90°/5 mm., $100°/10^{-3}$ mm. Sol. AcOH, C_6H_6. Less sol. EtOH, Et_2O, $CHCl_3$. Volatile in steam. Shows weak blue fluor.

Smith, Takamatsu, *J. Chem. Soc.*, 1881, **39**, 547.
Späth, *Monatsh.*, 1912, **33**, 1046.
Carter, van Loon, *J. Am. Chem. Soc.*, 1938, **60**, 1077.
Hey, Lawton, *J. Chem. Soc.*, 1940, 374.
Weizmann, Bergmann, Bograchov, *Chemistry and Industry*, 1940, 402.

Phenylnaphthalene-carboxylic Acid.
See β-Chrysenic Acid *and* Phenylnaphthoic Acid.

2-Phenylnaphthalene-1 : 2′-dicarboxylic Acid.
See Chrysodiphenic Acid.

1-Phenylnaphthalene-2 : 3-dicarboxylic Acid

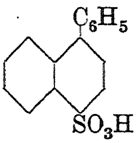


$C_{18}H_{12}O_4$ MW, 292

Leaflets from H_2O. At 255° ⟶ anhydride.
2-Me ester: $C_{19}H_{14}O_4$. MW, 306. Needles from EtOH or C_6H_6–ligroin. M.p. 207° decomp.
Di-Me ester: $C_{20}H_{16}O_4$. MW, 320. Leaflets from MeOH.Aq. M.p. 118–20° (121°). Sol. hot EtOH. Spar. sol. Et_2O.
2-Et ester: $C_{20}H_{16}O_4$. MW, 320. Needles from C_6H_6–ligroin. M.p. 207° decomp. Sol. most org. solvents. Insol. H_2O.
Di-Et ester: $C_{22}H_{20}O_4$. MW, 348. Plates from ligroin. M.p. 129–30°.
Anhydride: needles from C_6H_6–ligroin. M.p. 255° (257–9°). Sol. $CHCl_3$, AcOH, C_6H_6. Spar. sol. EtOH. Insol. ligroin.
Imide: needles from EtOH. M.p. 246°. Sol. EtOH, AcOH, C_6H_6.

Lanser, Halvorsen, *Ber.*, 1902, **35**, 1407.
Stobbe, *Ber.*, 1907, **40**, 3373.
Michael, Bucher, *Am. Chem. J.*, 1898, **20**, 95.
Pfeiffer, Moller, *Ber.*, 1907, **40**, 3841.

1-Phenylnaphthalene-4-sulphonic Acid

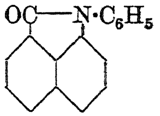


$C_{16}H_{12}O_3S$ MW, 284

Needles from C_6H_6. M.p. 165–7°.
Anilide: cryst. from C_6H_6–pet. ether. M.p. 167°. Very sol. C_6H_6.

v. Braun, Anton, *Ber.*, 1934, **67**, 1053.

***N*-Phenylnaphthastyril**



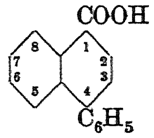
$C_{17}H_{11}ON$ MW, 245

Yellow needles from ligroin. M.p. 104–5°. Mod. sol. usual org. solvents, hot conc. HCl. Sol. hot, insol. cold NaOH.Aq.

I.G., F.P., 778,254, (*Chem. Abstracts*, 1935, **29**, 4600).
Rule, Turner, *J. Chem. Soc.*, 1935, 318.

2-Phenyl-1-naphthoic Acid.
See β-Chrysenic Acid.

4-Phenyl-1-naphthoic Acid (4-*Phenyl-naphthalene*-1-*carboxylic acid*)



$C_{17}H_{12}O_2$ MW, 248

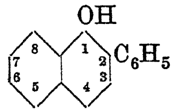
Cryst. from AcOH.Aq. M.p. 172–3°.
Nitrile: $C_{17}H_{11}N$. MW, 229. Cryst. from C_6H_6. M.p. 114–15°.

v. Braun, Anton, *Ber.*, 1934, **67**, 1054.

5-Phenyl-1-naphthoic Acid (5-*Phenyl-naphthalene*-1-*carboxylic acid*).
Cryst. from AcOH. M.p. 229°.
Nitrile: cryst. from C_6H_6. M.p. 73–7°.

v. Braun, Anton, *Ber.*, 1934, **67**, 1054.

2-Phenyl-1-naphthol (*1-Hydroxy-2-phenylnaphthalene*)



$C_{16}H_{12}O$ MW, 220

M.p. 67°.

Benzoate: m.p. 111°.

Lüttringhaus, Wagner-v.Sääf, *Ann.*, 1945, **557**, 25.

3-Phenyl-1-naphthol (*4-Hydroxy-2-phenylnaphthalene*).

Brownish needles from EtOH.Aq. M.p. 100–1°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. boiling H_2O.

Ruhemann, *J. Chem. Soc.*, 1910, **97**, 461.

4-Phenyl-1-naphthol (*4-Hydroxy-1-phenylnaphthalene*).

Needles from C_6H_6–pet. ether. M.p. 140°.

v. Braun, Anton, *Ber.*, 1934, **67**, 1053.
Borsche, *Ann.*, 1936, **526**, 1.

6-Phenyl-2-naphthol (*6-Hydroxy-2-phenylnaphthalene*).

Colourless plates from C_6H_6–ligroin. M.p. 175–6°.

Me ether: pale yellow platelets from EtOH. M.p. 148°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

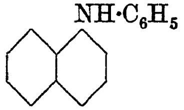
7-Phenyl-2-naphthol (*7-Hydroxy-2-phenylnaphthalene*).

Pale yellow needles from C_6H_6–ligroin. M.p. 156°.

Me ether: white plates from EtOH.Aq. or ligroin. M.p. 80°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

N-Phenyl-1-naphthylamine



$C_{16}H_{13}N$ MW, 219

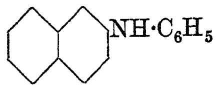
Prisms or needles from EtOH, leaflets from ligroin. M.p. 62°. B.p. 335°/528 mm., 226°/8 mm. (200°/10 mm.). Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Sols. show blue fluor. Heat of comb. C_v 2017 Cal. Conc. $H_2SO_4 + HNO_3 \longrightarrow$ blue col. $\longrightarrow$ green $\longrightarrow$ brown on heating. Rubber vulcanisation accelerator.

N-*Acetyl*: cryst. from EtOH. M.p. 115°. Sol. EtOH, $CHCl_3$, C_6H_6. Spar. sol. Et_2O.

N-*Benzoyl*: cryst. from EtOH. M.p. 152°. Sol. EtOH, Et_2O, C_6H_6.

Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 17.
Knoll, D.R.P., 241,853, (*Chem. Zentr.*, 1912, I, 178).
Streiff, *Ann.*, 1881, **209**, 154.
Hodgson, Marsden, *J. Soc. Chem. Ind.*, 1939, **58**, 154.

N-Phenyl-2-naphthylamine



$C_{16}H_{13}N$ MW, 219

Needles from MeOH. M.p. 108° (102–4°). B.p. 395–399·5°, 237°/13 mm. Mod. sol. EtOH, Et_2O, AcOH, C_6H_6. Sols. show blue fluor. Heat of comb. C_v 2007 Cal. Rubber vulcanisation accelerator.

N-*Acetyl*: cryst. from Et_2O. M.p. 93°. Sol. EtOH, Et_2O, AcOH, C_6H_6.

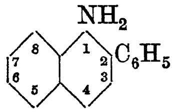
N-*Benzoyl*: needles from EtOH. M.p. 136° (147–8°). Sol. C_6H_6. Spar. sol. EtOH.

N-*Me*: $C_{17}H_{15}N$. MW, 233. M.p. 52–3°.

N-*Et*: $C_{18}H_{17}N$. MW, 247. Leaflets. M.p. 58°.

Streiff, *Ann.*, 1881, **209**, 157.
Cherntzov, Drozdov, *Chem. Abstracts*, 1935, **29**, 2529.
Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 17.
Knoll, D.R.P. 241,853, (*Chem. Zentr.*, 1912, I, 178).
Gershzon, *Chem. Abstracts*, 1944, **38**, 1479.

2-Phenyl-1-naphthylamine



$C_{16}H_{13}N$ MW, 219

Light brown needles from pet. ether. M.p. 104°.

N-*Acetyl*: white plates from EtOH.Aq. M.p. 234°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

4-Phenyl-1-naphthylamine.

Cryst. M.p. 73–4°.

N-*Acetyl*: m.p. 167–8°.

Veselý, Štursa, *Chem. Zentr.*, 1933, II, 3850.

6-Phenyl-1-naphthylamine.

Needles from pet. ether. M.p. 142–3°.

N-*Acetyl*: needles from EtOH. M.p. 131°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

7-Phenyl-1-naphthylamine.

Needles from EtOH.Aq. M.p. 94°.

N-*Acetyl*: small plates from EtOH. M.p. 203°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

6-Phenyl-2-naphthylamine.

Pale brown needles from pet. ether. M.p. 132°.

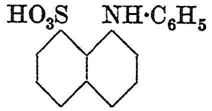
N-*Acetyl*: plates from EtOH.Aq. M.p. 199°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

Phenylnaphthylamine-carboxylic Acid.

See Naphthylanthranilic Acid.

N-Phenyl-1-naphthylamine-8-sulphonic Acid (*Phenyl-peri acid, 1-anilinonaphthalene-8-sulphonic acid*)



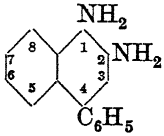
$C_{16}H_{13}O_3NS$ MW, 299

Leaflets. Spar. sol. H_2O. Intermediate for azo dyes.

Na salt: needles + H_2O. Very sol. H_2O.

Bayer, D.R.P., 70,349.
Kalle, D.R.P., 170,630, (*Chem. Zentr.*, 1906, II, 473).
A.G.F.A., D.R.P., 158,923, (*Chem. Zentr.*, 1905, I, 909).
Hodgson, Marsden, *J. Soc. Chem. Ind.*, 1939, **58**, 154.

4-Phenyl-1 : 2-naphthylenediamine



$C_{16}H_{14}N_2$ MW, 234

Cryst. M.p. 100–1°.

Veselý, Štursa, *Chem. Zentr.*, 1933, II, 3850.

2-Phenyl-1 : 3-naphthylenediamine.

Plates from MeOH. M.p. 116°. Sol. EtOH, C_6H_6. Spar. sol. cold Et_2O. Insol. pet. ether. Turns red in air.

1 : 3-N-*Di-Me*: $C_{18}H_{18}N_2$. MW, 262. *Cis*: Dimorphous. Needles or prisms from EtOH. M.p. 170°. Needles are stable between 20° and 80°. Prisms formed below 17° and above 80°. *Trans*: cryst. from EtOH. M.p. 159–60°. *Acetyl*: prisms from EtOH. M.p. 203°. *Diacetyl*: prisms from MeOH.Aq. M.p. 207–8°. p-*Toluenesulphonyl*: needles from EtOH. M.p. 219–20°. *Di*-p-*toluenesulphonyl*: plates from Py. M.p. 305°.

N : N-*Tri-Me*: $C_{19}H_{20}N_2$. MW, 276. Needles from EtOH. M.p. 104–5°. *Acetyl*: needles from EtOH. M.p. 178°.

N : N-*Tetra-Me*: $C_{20}H_{22}N_2$. MW, 290. Prisms from EtOH. M.p. 122°.

3-N-*Acetyl*: plates from EtOH or C_6H_6. M.p. 220°.

1 : 3-N-*Diacetyl*: needles. M.p. 267°.

1 : 3-N-*Di*-p-*toluenesulphonyl*: *cis*, dimorphous prisms from MeOH. M.p.s 203–5° and 188–9°. *Trans*: needles from MeOH. M.p. 173–5°.

N : N-*Tri*-p-*toluenesulphonyl*: needles from MeOH. M.p. 153–4°.

Atkinson, Thorpe, *J. Chem. Soc.*, 1906, **89**, 1934.
Atkinson, Ingham, Thorpe, *J. Chem. Soc.*, 1907, **91**, 589.
Lees, Thorpe, *ibid.*, 1296.
Gibson, Kentish, Simonsen, *J. Chem. Soc.*, 1928, 2131.
Lesslie, Turner, *J. Chem. Soc.*, 1929, 1512.

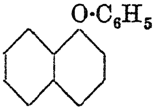
2-Phenyl-1 : 4-naphthylenediamine.

Brownish needles. M.p. 100–1°.

N-*Diacetyl*: small white needles from hot AcOH. M.p. 320°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 374.

Phenyl 1-naphthyl Ether (*1-Phenoxynaphthalene*)



$C_{16}H_{12}O$ MW, 220

Prisms from Et_2O–EtOH. M.p. 55–6°. B.p. 349·5°. Sol. Et_2O, $CHCl_3$, C_6H_6. Mod. sol. EtOH, ligroin.

Ullmann, Sponagel, *Ann.*, 1906, **350**, 90.
Fritzsche, D.R.P., 269,543, (*Chem. Zentr.*, 1914, I, 591).

Phenyl 2-naphthyl Ether (*2-Phenoxynaphthalene*).

Needles from Et_2O–EtOH. M.p. 46°. B.p. 335·5°/753 mm. Sol. Et_2O, $CHCl_3$, AcOH. Insol. H_2O.

Ullmann, Sponagel, *Ann.*, 1906, **350**, 90.
Fritzsche, D.R.P. 269,543, (*Chem. Zentr.*, 1914, I, 591).

***sym.*-Phenyl-1-naphthylhydrazine** (*Benzenehydrazo-α-naphthalene*)

$C_6H_5·NH·NH·C_{10}H_7$

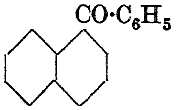
$C_{16}H_{14}N_2$ MW, 234

Leaflets. M.p. 125°.

Diacetyl: m.p. 264°.

Nietzki, Zehntner, *Ber.*, 1883, **26**, 144.

Phenyl 1-naphthyl Ketone (*1-Benzoylnaphthalene*)



$C_{17}H_{12}O$ MW, 232

Prisms from EtOH. M.p. 75·5–76°. B.p. 385°, 225°/12 mm., 222°/8 mm. Sol. 41 parts EtOH at 12°. Diamagnetic.

Oxime: exists in two forms. (i) Cryst. from EtOH. M.p. 161°. (ii) Cryst. from EtOH. M.p. 127°.

Imide: m.p. 68–9°. B.p. 181·5°/4·5 mm.

2 : 4-*Dinitrophenylhydrazone*: exists in two forms. α-. Red microscopic cryst. M.p. 246–7°. β-. Orange-yellow cryst. M.p. 243–4°. Mixture. M.p. 220°.

Moureu, Mignonac, *Compt. rend.*, 1913, **156**, 1806.
Betti, Poccianti, *Gazz. chim. ital.*, 1915, **45**, i, 374.
Caille, *Compt. rend.*, 1911, **153**, 393.
Szperl, *Chem. Zentr.*, 1930, I, 3436.
Given, Hammick, *J. Chem. Soc.*, 1947, 1237.
Nunn, Henze, *J. Org. Chem.*, 1947, **12**, 540.
Baddeley, *J. Chem. Soc.*, 1949, 99, *Suppl.*

Phenyl 2-naphthyl Ketone (*2-Benzoylnaphthalene*).

Needles from EtOH. M.p. 82°. B.p. 398°/754 mm., 225°/8 mm. Sol. 49 parts EtOH at 12°. Paramagnetic.

Oxime: exists in two forms. (i) Needles from EtOH. M.p. 174°. (ii) Cryst. from EtOH. M.p. 157°.

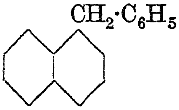
Semicarbazone: cryst. from EtOH. M.p. 175°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Insol. H_2O.

2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH–AcOH. M.p. 257–8°.

Picrate: m.p. 112–13°.

Lecher, *Ber.*, 1913, **46**, 2667; D.R.P., 281,802, (*Chem. Zentr.*, 1915, I, 281).
Poccianti, *Gazz. chim. ital.*, 1915, **45**, ii, 114.
Barbot, *Bull. soc. chim.*, 1930, **47**, 1314.
Orchin, Woolfolk, Reggel, *J. Am. Chem. Soc.*, 1949, **71**, 1126.
Pfeiffer, Jenning, Stöcker, *Ann.*, 1949, **563**, 73.
Baddeley, *J. Chem. Soc.*, 1949, 99, *Suppl.*

Phenyl-1-naphthylmethane (*1-Benzylnaphthalene*)



$C_{17}H_{14}$ MW, 218

M.p. 58·5°. B.p. 350°, 217–20°/20 mm. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. D^{17} 1·166. Dil. HNO_3 ⟶ phenyl 1-naphthyl ketone.

Picrate: yellow needles. M.p. 100–1°.

Nenitzescu, Isăcescu, Ionescu, *Ann.*, 1931, **491**, 217.
Miquel, *Bull. soc. chim.*, 1876, **26**, 2.
Roux, *Ann. chim. phys.*, 1887, **12**, 326.
Lecher, D.R.P., 281,802, (*Chem. Zentr.*, 1915, I, 281).
Dziewoński, Dziecielewski, *Chem. Zentr.*, 1928, I, 57.
Gasopoulos, *Chem. Zentr.*, 1933, II, 43.
Bradlow, Vander Werf, *J. Am. Chem. Soc.*, 1947, **69**, 1254.
Monacelli, Hennion, *J. Am. Chem. Soc.*, 1941, **63**, 1722.

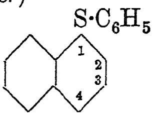
Phenyl-2-naphthylmethane (*2-Benzylnaphthalene*).

Thick colourless prisms from EtOH. M.p. 58°. B.p. 350°. Sol. EtOH, C_6H_6. D^0 1·176. Dil. HNO_3 ⟶ phenyl 2-naphthyl ketone.

Picrate: golden-yellow needles from EtOH. M.p. 94–5°.

Roux, *Ann. chim. phys.*, 1887, **12**, 326.
Vincent, Roux, *Bull. soc. chim.*, 1883, **40**, 165.
Dziewoński, Wodelski, *Chem. Zentr.*, 1933, I, 774.
Orchin, Woolfolk, Reggel, *J. Am. Chem. Soc.*, 1949, **71**, 1126.
Borsche, Hofmann, Kühn, *Ann.*, 1943, **554**, 23.

Phenyl 1-naphthyl sulphide (*1-Thionaphthol phenyl ether*)



$C_{16}H_{12}S$ MW, 236

Prisms from EtOH.Aq. M.p. 41·8°. B.p. 255–6°/43 mm., 220–5°/11 mm. D_4^{15} 1·167. Mod. sol. EtOH, Et_2O.

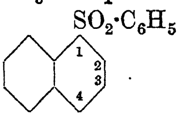
Wuyts, *Bull. soc. chim.*, 1906, **35**, 167.
Bourgeois, *Ber.*, 1895, **28**, 2327.

Phenyl 2-naphthyl sulphide (*2-Thionaphthol phenyl ether*).

Needles or leaflets from EtOH. M.p. 51·8°. B.p. 226°/11 mm.

Bourgeois, *Ber.*, 1895, **28**, 2327.

Phenyl 1-naphthyl sulphone



$C_{16}H_{12}O_2S$ MW, 268

Cryst. from EtOH. M.p. 99·5–100·5°. Very sol. hot EtOH, Et_2O, AcOH, C_6H_6.

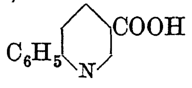
Michael, Adair, *Ber.*, 1877, **10**, 585.

Phenyl 2-naphthyl sulphone.

Needles from EtOH or Et_2O. M.p. 115–16°.

Michael, Adair, *Ber.*, 1877, **10**, 585.

6-Phenylnicotinic Acid (*6-Phenylpyridine-3-carboxylic acid*)



$C_{12}H_9O_2N$ MW, 199

Needles from H_2O. M.p. 232–3° (sinters at 220°). Sol. EtOH, Et_2O. Spar. sol. H_2O, AcOH.

Anilide: leaflets from EtOH. M.p. 199°.

Bemary, Paille, *Ber.*, 1924, **57**, 832.

Phenylnitramine (*N-Nitroaniline, nitraminobenzene*)

$$C_6H_5\cdot NH\cdot NO_2 \rightleftharpoons C_6H_5\cdot N{:}N(\rightarrow O)\cdot OH$$

aci-form

$C_6H_6O_2N_2$ MW, 138

Pearly leaflets from pet. ether. M.p. 46–46·5°. Very sol. most org. solvents. Mod. sol. H_2O. Spar. sol. pet. ether. $k = 1{\cdot}7 \times 10^{-5}$ at 18°, $2{\cdot}3 \times 10^{-5}$ at 25°. Explodes on strong heating. Warm dil. min. acids ⟶ *o*- and *p*-nitroaniline. Mod. volatile in steam.

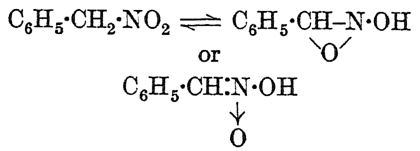
N-*Me*: *see* Methylphenylnitramine.

aci-*Me ether*: $C_7H_8O_2N_2$. MW, 152. Yellow oil with odour of heliotrope. Decomp. with explosion on warming.

Bamberger, *Ber.*, 1893, **26**, 485; 1894, **27**, 362, 915.
Degner, v. Pechmann, *Ber.*, 1897, **30**, 647.

Phenyl nitrobenzyl Ketone.
See Nitrodeoxybenzoin.

Phenylnitromethane (ω-*Nitrotoluene*)



aci-form

$C_7H_7O_2N$ MW, 137

Yellow liq. B.p. 225–7°, 141–2°/35 mm., 118–19°/16 mm. D_0^0 1·1756, D_0^{20} 1·1598. n_D^{20} 1·53230. Gives no col. with $FeCl_3$. Alkalis ⟶ *aci*-form. Electrolytic reduction ⟶ benzylamine.

aci-*Form*: phenylisonitromethane. Cryst. from Et_2O–pet. ether. M.p. 84°. Sol. EtOH, Et_2O. Mod. sol. C_6H_6. Insol. pet. ether. Sol. Na_2CO_3.Aq. $FeCl_3$ ⟶ intense reddish-brown col. *NH_4 salt*: powder. M.p. 89–90°.

Hantzsch, Schultze, *Ber.*, 1896, **29**, 700.
Konowalow, *Ber.*, 1895, **28**, 1861.
Hollemann, *Rec. trav. chim.*, 1894, **13**, 405.
Wislicenus, Grützner, *Ber.*, 1909, **42**, 1932.
Schorygin, Ssokolowa, *Chem. Zentr.*, 1930, II, 2637.
Black, Babers, *Organic Syntheses*, Collective Vol. I, 512.

S-Phenyl-4-nitrothiosalicylic Acid.
See 5-Nitrodiphenylsulphide-2-carboxylic Acid.

Phenyl 6-nitro-*m*-tolyl Ether.
See 6-Nitro-3-methyldiphenyl Ether.

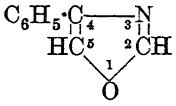
Phenyl nitrotolyl Ketone.
See Nitromethylbenzophenone.

Phenyl nitrotolyl sulphide.
See Nitromethyldiphenyl sulphide.

Phenyl nitrotolyl sulphone.
See Nitromethyldiphenyl sulphone.

Phenyloctane.
See Octylbenzene.

4-Phenyloxazole



C_9H_7ON MW, 145

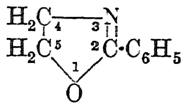
Thick liq. M.p. 6°. B.p. 220–2°.
B,HCl: plates. M.p. 80°.

Lewy, *Ber.*, 1887, **20**, 2578.
Blümlein, *Ber.*, 1884, **17**, 2580.

5-Phenyloxazole.
Cryst. M.p. 41–2°.
$B_2,AuCl_3$: orange needles from H_2O. M.p. 149–50°.
Chloroplatinate: long yellow needles. Does not decompose below 275°. Spar. sol. H_2O.

Bachstez, *Ber.*, 1914, **47**, 3165.

2-Phenyl-2-oxazoline (2-*Phenyl*-4:5-*dihydro-oxazole*)

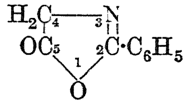


C_9H_9ON MW, 147

B.p. 246–8° (242–3°). Misc. with EtOH, Et_2O. Spar. sol. H_2O.
Picrate: yellow needles. M.p. 178–9°. Spar. sol. most solvents.

Gabriel, Neumann, *Ber.*, 1892, **25**, 2385.
Gabriel, Stelzner, *Ber.*, 1895, **28**, 2933.
Wenker, *J. Am. Chem. Soc.*, 1935, **57**, 1080.

2-Phenyl-5(4)-oxazolone

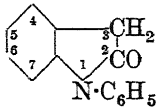


$C_9H_7O_2N$ MW, 161

Cryst. from Et_2O. M.p. 91°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O, pet. ether.

Karrer, Bussmann, *Helv. Chim. Acta*, 1941, **24**, 645.
Karrer, Widmer, *Helv. Chim. Acta*, 1925, **8**, 203.

1-Phenyloxindole



$C_{14}H_{11}ON$ MW, 209

Leaflets from H_2O or ligroin. M.p. 121°. Sol. EtOH, Et_2O. Spar. sol. H_2O.

Stollé, *Ber.*, 1914, **47**, 2120; D.R.P., 335,673, (*Chem. Abstracts*, 1923, **17**, 1802).

3-Phenyloxindole.
Reddish leaflets from EtOH. M.p. 183° (185–7°). Sol. EtOH, C_6H_6. Insol. acids. Sol. KOH.Aq.
N-*Acetyl*: cryst. from EtOH.Aq. M.p. 103°.

Brunner, *Monatsh.*, 1897, **18**, 547.
Meisenheimer, Camparter, *Ber.*, 1924, **57**, 297.

7-Phenylpalmitic Acid

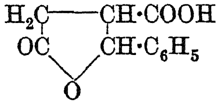
$$CH_3\cdot[CH_2]_7\cdot\overset{C_6H_5}{\overset{|}{C}H}\cdot[CH_2]_6\cdot COOH$$

$C_{22}H_{36}O_2$ MW, 332

B.p. 190–5°/0·12 mm. D_{25}^{25} 0·9417. n_D^{20} 1·4912.
Et ester: $C_{24}H_{40}O_2$. MW, 360. B.p. 174–80°/0·13 mm. D_{25}^{25} 0·9194. n_D^{20} 1·4808.
p-*Bromophenacyl ester*: m.p. 77–8°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2515.

γ-Phenylparaconic Acid (*3-Phenyl-γ-butyrolactone-2-carboxylic acid*)



$C_{11}H_{10}O_4$ MW, 206

d-.

Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. anhyd. 134°. Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O, hot C_6H_6. Insol. CS_2, pet. ether. $[\alpha]_D^{20}$ + 64·3° in EtOH.

l-.

Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. anhyd. 134°. Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O, hot C_6H_6. Insol. CS_2, pet. ether. $[\alpha]_D^{20}$ − 65·3° in EtOH.

dl-.

Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. 99°, anhyd. 121°. $k = 4{\cdot}80 \times 10^{-4}$ at 20°. Heat of comb. of hydrate, C_v 1195·9 Cal.

Me ester: $C_{12}H_{12}O_4$. MW, 220. Cryst. from Et_2O. M.p. 69–70°. B.p. 211°/14 mm.

Et ester: $C_{13}H_{14}O_4$. MW, 234. Oil with aromatic odour. B.p. 241–2°/52 mm., 224°/25 mm.

Barbier, Locquin, *Bull. soc. chim.*, 1912, **13**, 233.
Fittig, Jehl, *Ann.*, 1904, **330**, 345.

5-Phenyl-1 : 2-pentadiene (*γδ-Pentadienylbenzene*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}C{:}CH_2$

$C_{11}H_{12}$ MW, 144

B.p. 76–7°/7 mm. D_4^{20} 0·9169. n_D^{20} 1·5400.

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **55**, 2816.

1-Phenyl-1 : 3-pentadiene (*αγ-Pentadienylbenzene*)

$CH_3{\cdot}CH{:}CH{\cdot}CH{:}CH{\cdot}C_6H_5$

$C_{11}H_{12}$ MW, 144

Viscous oil. B.p. 240–60°, 116°/16 mm. $D_4^{15\cdot2}$ 0·9325. $n_D^{15\cdot2}$ 1·6000.

Auwers, Eisenlohr, *J. prakt. Chem.*, 1911, **84**, 43.
Pestemer, Wiligot, *Monatsh.*, 1935, **66**, 123.
Koelsch, Boekelheide, *J. Am. Chem. Soc.*, 1944, **66**, 412.
Emerson, *J. Org. Chem.*, 1945, **10**, 464.

2-Phenyl-1 : 3-pentadiene

$CH_3{\cdot}CH{:}CH{\cdot}C(C_6H_5){:}CH_2$

$C_{11}H_{12}$ MW, 144

B.p. 85–6°/15 mm. Readily polymerises.

Kuhn, Hoffer, *Ber.*, 1933, **66**, 1269.

3-Phenylpentandione-2 : 4.

See Phenylacetylacetone.

1-Phenylpentane.

See *n*-Amylbenzene.

2-Phenylpentane (*ω-Methylethyltoluene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_3$

$C_{11}H_{16}$ MW, 148

B.p. 191–3°. D_4^{21} 0·8594. n_D^{21} 1·4875.

Klages, *Ber.*, 1902, **35**, 3509.

3-Phenylpentane (*Diethylphenylmethane, ω-diethyltoluene*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_2{\cdot}CH_3$

$C_{11}H_{16}$ MW, 148

B.p. 180° (187°), 83–5°/22 mm., 38–41°/0·7 mm. D_4^{15} 0·8755. n_D^{25} 1·4868.

Levene, Marker, *J. Biol. Chem.*, 1935, **108**, 417.

Phenylpentanol-1.

See Phenyl-*n*-amyl Alcohol.

1-Phenylpentanol-2.

See Propylbenzylcarbinol.

2-Phenylpentanol-2.

See Methylpropylphenylcarbinol.

4-Phenylpentanol-2.

See 4-Phenyl-*sec.*-*n*-amyl Alcohol.

5-Phenylpentanol-2.

See 5-Phenyl-*sec.*-*n*-amyl Alcohol.

1-Phenylpentanol-3 (*Ethyl-β-phenylethylcarbinol, γ-hydroxy-*n*-amylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$

$C_{11}H_{16}O$ MW, 164

d-.

M.p. 38°. B.p. 143°/19 mm. D_4^{20} 0·9687, D_4^{140} 0·8773. $[\alpha]_{5461}$ + 31·8° in EtOH.

Acid phthalate: viscous gum. $[\alpha]_{5461}$ + 42·9° in EtOH. *Strychnine salt*: m.p. 158°. $[\alpha]_{5461}$ − 38·7° in $CHCl_3$.

Formyl: b.p. 135°/15 mm. D_4^{20} 0·9980. n_{5461}^{20} 1·5003. $[\alpha]_{5461}^{20}$ − 13·93°.

Acetyl: b.p. 147°/19 mm. D_4^{20} 0·9829. n_{5461}^{20} 1·4920. $[\alpha]_{5461}^{18\cdot5}$ − 11·20°.

l-.

M.p. 38°. B.p. 143°/19 mm.

Acid phthalate: $[\alpha]_{5461}$ − 42·9° in EtOH. *Cinchonidine salt*: m.p. 154°. $[\alpha]_{5461}$ − 54·2° in EtOH.

Propionyl: b.p. 150°/14 mm. D_4^{20} 0·9731. n_{5461}^{20} 1·4878. $[\alpha]_{5461}^{20}$ + 22·47°.

dl.-

Liq. B.p. 143°/19 mm., 130°/15 mm.

Acid phthalate: prisms from AcOH. M.p. 74°.

Phenylurethane: m.p. 74°.

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1094.
Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

2-Phenylpentanol-3 (*Ethyl-α-phenylethylcarbinol*)

$CH_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CH(C_6H_5){\cdot}CH_3$

$C_{11}H_{16}O$ MW, 164

B.p. 134–7°/32 mm.

Tiffeneau, Lévy, *Bull. soc. chim.*, 1923, 33, 759.

3-Phenylpentanol-3 (*Diethylphenylcarbinol*)

$CH_3{\cdot}CH_2{\cdot}C(C_6H_5)(OH){\cdot}CH_2{\cdot}CH_3$

$C_{11}H_{16}O$ MW, 164

B.p. 223–4°/762 mm., 159–61°/150 mm., 109°/15 mm. D_4^{19} 0·9836. n_D^{20} 1·51655.

Klages, *Ber.*, 1903, **36**, 3692.
Gilman, Fothergill, Parker, *Rec. trav. chim.*, 1929, **48**, 748.

1-Phenylpentanone-2.
See Propyl benzyl Ketone.

1-Phenylpentanone-3 (*γ-Keto*-n-*amylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$

$C_{11}H_{14}O$ MW, 162

B.p. 244°/750 mm., 140°/14 mm. (128°/17 mm.). D_4^0 0·9793. n_D^{20} 1·50882.
Semicarbazone: m.p. 131–2° (82°, 100–35°). M.p. varies according to method of preparation.

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1096.
Maxim, *Ann. chim.*, 1928, **9**, 55.
Montagne, Roch, *Compt. rend.*, 1944, **218**, 679.

1-Phenylpentanone-4 (*5-Phenylpentanone-2, γ-acetopropylbenzene, δ-keto*-n-*amylbenzene*)

$CH_3{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_5$

$C_{11}H_{14}O$ MW, 162

Mobile liq. B.p. 128–30°/15 mm. n_D^{19} 1·510.
Semicarbazone: leaflets from MeOH. M.p. 127°.

Diels, Poetsch, *Ber.*, 1921, **54**, 1585.
Heilbron, Heslop, Irving, Wilson, *J. Chem. Soc.*, 1931, 1340.
Tiffeneau, Deux, *Compt. rend.*, 1942, **214**, 892.
Prévost, Robert, *Bull. soc. chim.*, 1944, **11**, 225.

2-Phenylpentanone-3 (*ω-Methyl-ω-propionyl-toluene*)

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}CH(C_6H_5){\cdot}CH_3$

$C_{11}H_{14}O$ MW, 162

B.p. 225–8° (222–5°). D^0 0·982.
Semicarbazone: m.p. 136°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.
Lévy, Tabart, *Bull. soc. chim.*, 1931, **49**, 1776.

2-Phenylpentanone-4 (*4-Phenylpentanone-2, β-acetoisopropylbenzene*)

$CH_3{\cdot}CO{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH_3$

$C_{11}H_{14}O$ MW, 162

B.p. 109–10°/11 mm. D_4^{20} 0·9708. n_D^{20} 1·51237.
Oxime: viscous liq. B.p. 160°/20 mm.
Semicarbazone: m.p. 145° (137°).

Rupe, Wild, *Ann.*, 1917, **414**, 124.
Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 260.

3-Phenylpentanone-2 (*ω-Ethylacetyltoluene*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CO{\cdot}CH_3$

$C_{11}H_{14}O$ MW, 162

B.p. 220–5° (215–20°). D^0 0·979. Does not form bisulphite comp.
Semicarbazone: m.p. 189–90°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.
Tiffeneau, Lévy, Jullien, *Bull. soc. chim.*, 1931, **49**, 1788.

1-Phenyl-1-pentene (*α-Phenyl-α-amylene, α-pentenylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{:}CH{\cdot}C_6H_5$

$C_{11}H_{14}$ MW, 146

B.p. 217° (202–5°), 94°/14 mm., 87·5°/9 mm. D_4^{22} 0·8782. n_D^{20} 1·5158 (1·5330).
Nitrosite: m.p. 121°.
Dibromide: m.p. 61°.

v. Braun, Köhler, *Ber.*, 1918, **51**, 79.
Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.
Lévy, Dvoleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1765.
Levina, Shcheglova, *J. Gen. Chem. U.S.S.R.*, 1941, **11**, 527, (*Chem. Abstracts*, 1941, **35**, 6936).

2-Phenyl-1-pentene (*β-Phenyl-α-amylene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}C(C_6H_5){:}CH_2$

$C_{11}H_{14}$ MW, 146

B.p. 198–202°. D^0 0·9138.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 357.

3-Phenyl-1-pentene (*γ-Phenyl-α-amylene, amenylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH{:}CH_2$

$C_{11}H_{14}$ MW, 146

B.p. 191·5° (173°), 71°/12 mm. D^{23} 0·8458. n^{21} 1·5030.

Dafert, *Monatsh.*, 1883, **4**, 621.
Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.

5-Phenyl-1-pentene (*ε-Phenyl-α-amylene, δ-pentenylbenzene*)

$C_6H_5·CH_2·CH_2·CH_2·CH:CH_2$

$C_{11}H_{14}$ MW, 146

B.p. 203–4°, 77–8°/10 mm. D_4^{20} 0·8889. n_D^{20} 1·5065.

v. Braun, Deutsch, Schmatloch, *Ber.*, 1912, **45**, 1255.

1-Phenyl-2-pentene (*α-Phenyl-β-amylene, β-pentenylbenzene*)

$CH_3·CH_2·CH:CH·CH_2·C_6H_5$

$C_{11}H_{14}$ MW, 146

B.p. 201°, 111°/30 mm., 97–8°/19 mm. $D_4^{16·2}$ 0·8884. $n_D^{16·2}$ 1·5089. Alc. KOH at 150° ⟶ 1-phenyl-1-pentene.

Auwers, Roth, Eisenlohr, *Ann.*, 1910, **373**, 284.
Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.
Arbuzov, Kuznetsov, *Compt. rend. acad. sci. U.R.S.S.*, 1943, **39**, 311, (*Chem. Abstracts*, 1945, **39**, 1146).

2-Phenyl-2-pentene (*β-Phenyl-β-amylene*)

$CH_3·CH_2·CH:C(C_6H_5)·CH_3$

$C_{11}H_{14}$ MW, 146

Oil. B.p. 203–5°, 90°/16 mm. $D_4^{26·5}$ 0·8950. $n_D^{26·5}$ 1·5196. Acid $KMnO_4$ ⟶ acetophenone. Na + EtOH ⟶ 2-phenylpentane.

Klages, *Ber.*, 1902, **35**, 3509.
Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 363.

3-Phenyl-2-pentene (*γ-Phenyl-β-amylene*)

$CH_3·CH_2·C(C_6H_5):CH·CH_3$

$C_{11}H_{14}$ MW, 146

B.p. 197–8°/753 mm., 92°/18 mm. D_4^{14} 0·9173. n_D^{15} 1·5266.

Nitrosochloride: needles. M.p. 117°.

Klages, *Ber.*, 1903, **36**, 3692.

5-Phenyl-2-pentene (*ε-Phenyl-β-amylene, γ-pentenylbenzene*)

$C_6H_5·CH_2·CH_2·CH:CH·CH_3$

$C_{11}H_{14}$ MW, 146

B.p. 203–4°.

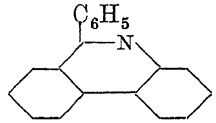
I.G., B.P. 315,312, (*Chem. Zentr.*, 1930, I, 2161).

Phenyl-peri Acid.
See N-Phenyl-1-naphthylamine-8-sulphonic Acid.

Phenylphenacylacetic Acid.
See 1-Phenyl-2-benzoylpropionic Acid.

Phenyl phenacyl Ketone.
See Dibenzoylmethane.

9-Phenylphenanthridine



$C_{19}H_{13}N$ MW, 255

Plates from EtOH. M.p. 106–7° (104–5°). B.p. above 400°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Mod. sol. hot H_2O. Spar. sol. ligroin. Volatile in steam. Salts easily hyd. by H_2O.

B,HCl: needles. M.p. 220°.

B,HCl,H₂O: cryst. from dil. HCl. M.p. 95–6°.

Nitrate: needles. M.p. 205°.

$B_2,H_2PtCl_6,2H_2O$: yellow needles. Decomp. about 300° without melting.

Picrate: yellow needles from EtOH. M.p. 246–7° decomp.

Pictet, Hubert, *Ber.*, 1896, **29**, 1183, 1187.
Gilman, Nelson, *J. Am. Chem. Soc.*, 1948, **70**, 3316.
Oxley, Short, *J. Chem. Soc.*, 1949, 449.
Short, Cymerman, B.P. 614,072, (*Chem. Abstracts*, 1949, **43**, 5050).

N-Phenyl-p-phenetidine.
See under 4-Hydroxydiphenylamine.

Phenylphenol.
See Hydroxydiphenyl.

Phenylphenylenediamine.
See Aminodiphenylamine.

Phenyl phenylethyl Ether.
See under 2-Phenylethyl Alcohol.

Phenyl phenylethyl Ketone.
See ω-Benzylacetophenone.

α-Phenyl-phenylglycine.
See α-Anilinophenylacetic Acid.

Phenylphosphine (*Phosphaniline*)

$C_6H_5PH_2$

C_6H_7P MW, 110

B.p. 160–1°. D^{15} 1·001. Oxidises very rapidly in air.

B,HI: needles. M.p. 138°. Decomp. by H_2O into its components.

Köhler, Michaelis, *Ber.*, 1877, **10**, 808.

Phenylphosphorous Acid

$C_6H_5·PO(OH)_2$

$C_6H_7O_3P$ MW, 158

Leaflets from H_2O. M.p. 158°. Sol. EtOH, Et_2O. Insol. C_6H_6. Stable in air.

Di-Me ester: $C_8H_{11}O_3P$. MW, 186. B.p. 247°, 101–2°/15 mm. D_0^{20} 1·0849. n_D^{20} 1·5280.

Et ester: $C_8H_{11}O_3P$. MW, 186. Syrup. Decomp. on dist. *Anilide*: m.p. about 105°. Hygroscopic.

Di-Et ester: $C_{10}H_{15}O_3P$. MW, 214. Viscous liq. B.p. 267°.

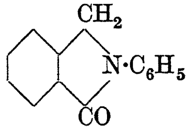
Phenyl ester: $C_{12}H_{11}O_3P$. MW, 234. Needles from EtOH.Aq. M.p. 57°. *Anilide*: yellow cryst. M.p. 83°. B.p. 235°/25 mm.
Diphenyl ester: $C_{18}H_{15}O_3P$. MW, 310. Needles from EtOH.Aq. M.p. 63·5°.
Dichloride: $C_6H_5OCl_2P$. MW, 195. Viscous liq. B.p. 258°. D^{20} 1·375.
Diamide: $C_6H_9ON_2P$. MW, 156. Leaflets from EtOH. M.p. 189°.
Anhydride: phosphinobenzene. $C_6H_5O_2P$. MW, 140. Cryst. powder from C_6H_6. M.p. 100°.
Anilide: m.p. 125°.
Dianilide: needles from EtOH. M.p. 211°.
Di-p-*toluidide*: needles from EtOH. M.p. 223°.
Di-phenylhydrazide: needles from EtOH. M.p. 175°.
Aniline salt: needles. M.p. 212°.

Arbusow, *Chem. Zentr.*, 1936, I, 543.
Michaelis, *Ann.*, 1876, **181**, 305,335; 1896, **293**, 217.
Michaelis, Köhler, *Ber.*, 1876, **9**, 521.

Phenylphthalic Acid.
See Diphenyl-dicarboxylic Acid.

N-Phenylphthalimide.
See Phthalanil.

N-Phenylphthalimidine (*Phthalidanil*)

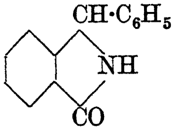


$C_{14}H_{11}ON$ MW, 209

Leaflets from EtOH. M.p. 162–3° (160°). Sol. $CHCl_3$, C_6H_6. Spar. sol. Et_2O. Very spar. sol. boiling H_2O. Insol. dil. acids and alkalis. CrO_3 in AcOH⟶phthalanil. Alk. $KMnO_4$⟶ phthalanilic acid.
Anil: needles from Et_2O. M.p. 142–3°. *Hydrochloride*: m.p. 237–8°. B_2,H_2PtCl_6: reddish-yellow cryst. from H_2O. M.p. 212-13° decomp.

Rowe, Levin, Burns, Davies, Tepper, *J. Chem. Soc.*, 1926, 704.
Fischer, Wolter, *J. prakt. Chem.*, 1910, **80**, 110.
Thiele, Schneider, *Ann.*, 1909, **369**, 297.
Graebe, Pictet, *Ann.*, 1888, **247**, 306.
Peters, Rowe, Brodrick, *J. Chem. Soc.*, 1948, 1249.

3-Phenylphthalimidine

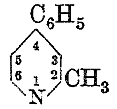


$C_{14}H_{11}ON$ MW, 209

Cryst. from 75% AcOH. M.p. 218–20°, solidifies at 230°, finally melting at 295–8° decomp. Sol. MeOH, EtOH. Spar. sol. Et_2O. Insol. pet. ether.
N-*Acetyl*: cryst. from AcOH. M.p. 154–5°.

Rose, *J. Am. Chem. Soc.*, 1911, **33**, 390.
Pfeiffer, Jaensch, *J. prakt. Chem.*, 1941, **159**, 241.

4-Phenyl-α-picoline (2-*Methyl*-4-*phenylpyridine*)



$C_{12}H_{11}N$ MW, 169

Liq. resembling diphenylamine in odour. B.p. 280–90°. Volatile in steam. Turns brown in air.
B,HClO_4: m.p. 142–3°.
B_2,H_2PtCl_6: orange-yellow needles. M.p. 211–13°.
$B,HAuCl_4$: m.p. 161–3°.
Picrate: yellow needles. M.p. 210–13° (203°).

Gohdes, *J. prakt. Chem.*, 1929, **123**, 184.

6-Phenyl-α-picoline (2-*Methyl*-6-*phenylpyridine*).
Liq. B.p. 280–1°. Volatile in steam.
$B,HAuCl_4$: yellow needles. M.p. 150–1°. Spar. sol. cold H_2O.
B_2,H_2PtCl_6,H_2O: reddish needles from H_2O. M.p. 200° decomp.
Picrate: yellow needles. M.p. 135°.

Scholtz, *Ber.*, 1895, **28**, 1726.

6-Phenyl-α-picoline-3-carboxylic Acid (2-*Methyl*-6-*phenylnicotinic acid*)

$C_{13}H_{11}O_2N$ MW, 213

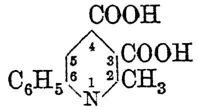
Cryst. from EtOH. M.p. 196°. Sol. Me_2CO, AcOEt. Spar. sol. H_2O, Et_2O, C_6H_6, ligroin. Readily sol. dil. acids.
B,HCl: m.p. 288°.
Et ester: $C_{15}H_{15}O_2N$. MW, 241. M.p. 46–46·5°. B.p. 185°/13 mm., 160–1°/2 mm. *Chloroplatinate*: m.p. 196°.
Methylbetaine: m.p. 240° decomp. *B_2,H_2SO_4*: prisms from EtOH. M.p. 223–4°. *$B,HAuCl_4$*: yellow needles. M.p. 160–1°.

Mumm, Neumann, *Ber.*, 1926, **59**, 1620.
Späth, Burger, *Monatsh.*, 1928, **49**, 265.
Nienburg, *Ber.*, 1935, **68**, 1475.

6-Phenyl-α-picoline-4-carboxylic Acid (2-*Methyl*-6-*phenylisonicotinic acid*).
Cryst. from EtOH. M.p. 272°. Sol. EtOH, Me_2CO, AcOEt. Spar. sol. H_2O, Et_2O, C_6H_6, ligroin. Sol. dil. HCl.
Et ester: b.p. 194°/16 mm. Solidifies to a mass of needles.

Mumm, Neumann, *Ber.*, 1926, **59**, 1621.

6-Phenyl-α-picoline-3 : 4-dicarboxylic Acid (2-*Methyl*-6-*phenylcinchomeronic acid*)



$C_{14}H_{11}O_4N$ MW, 257

Cryst. from EtOH. M.p. 217°. Sol. H_2O, EtOH, AcOH, HCl. Insol. Et_2O, AcOEt, Me_2CO, $CHCl_3$, C_6H_6.

3-*Et ester*: $C_{16}H_{15}O_4N$. MW, 285. Needles from ligroin. M.p. 145°. Sol. Me_2CO, AcOEt. Spar. sol. EtOH, Et_2O, C_6H_6, ligroin. Prac. insol. H_2O.

4-*Et ester*: needles from AcOEt. M.p. 185°. Mod. sol. Et_2O, AcOEt, C_6H_6, ligroin. Spar. sol. H_2O.

Di-Et ester: $C_{18}H_{19}O_4N$. MW, 313. Needle: from AcOEt. M.p. 73°. Sol. EtOH, Et_2Os Me_2CO, $CHCl_3$, C_6H_6. Mod. sol. ligroin. *Picrate*, m.p. 83°.

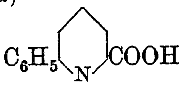
4-*Amide*: $C_{14}H_{12}O_3N_2$. MW, 256. Cryst. from EtOH. M.p. 199°. Spar. sol. org. solvents.

Anhydride: $C_{14}H_9O_3N$. MW, 239. Needles from C_6H_6. M.p. 196°. Sol. Et_2O, AcOEt, C_6H_6, ligroin.

Imide: $C_{14}H_{10}O_2N_2$. MW, 238. Needles from Me_2CO. M.p. 249°.

Mumm, Böhme, *Ber.*, 1921, **54**, 731.
Mumm, Neumann, *Ber.*, 1926, **59**, 1616.

6-Phenylpicolinic Acid (6-*Phenylpyridine*-2-*carboxylic acid*)

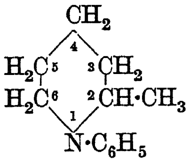


$C_{12}H_9O_2N$ MW, 199

Needles from H_2O. M.p. 109°. Very sol. EtOH. Heat at 190–200° ⟶ 2-phenylpyridine. $FeSO_4$ in H_2O ⟶ red col.

Scholtz, *Ber.*, 1895, **28**, 1728.

N-Phenyl-α-pipecoline (2-*Methyl*-N-*phenylpiperidine*)



$C_{12}H_{17}N$ MW, 175

B.p. 256·5–257°/710 mm., 143°/20 mm. n_D^{17} 1·55428. Sol. EtOH, Et_2O. Very spar. sol. H_2O.

B_2,H_2PtCl_6: orange-yellow needles. Decomp. at 212°.

Picrate: yellow prisms from EtOH. M.p. 167–8° (162°, 157–8°).

Methiodide: cryst. powder from EtOH–Et_2O. M.p. 145°.

v. Braun, Sobecki, *Ber.*, 1911, **44**, 1045.
Lipp, *Ann.*, 1896, **289**, 245.
Paul, *Bull. soc. chim.*, 1938, **5**, 919.

6-Phenyl-α-pipecoline (2-*Methyl*-6-*phenylpiperidine*).

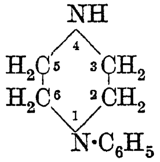
B.p. 112–14°/12 mm. D_4^{25} 0·9096. n_D^{25} 1·4882. Two inactive stereoisomeric forms are known:

α-.
B,HCl: m.p. 191–2°. Sol. Me_2CO.
B_2,H_2PtCl_6: m.p. 236°.

β-.
B.p. 117·5–118°/11 mm.
B,HCl: cryst. from Me_2CO. M.p. 225°.
B_2,H_2PtCl_6: m.p. 257°.

Adkins, Kuick, Farlow, Wojcik, *J. Am. Chem. Soc.*, 1934, **56**, 2425.
Singer, McElvain, *J. Am. Chem. Soc.*, 1935, **57**, 1137.
Meisenheimer, Stratman, Theilacker, *Ber.*, 1932, **65**, 423.

N-Phenylpiperazine



$C_{10}H_{14}N_2$ MW, 162

Oil. B.p. 156–7°/10 mm. D_4^{14} 1·0725. n_D 1·59053.

B,HCl: m.p. 247° decomp.
B,HBr: m.p. 250–2°.
4-*Acetyl*: m.p. 96°. *B,HCl*: m.p. 213–14°.
4-*Benzoyl*: m.p. 96–7°. *B,HCl*: m.p. 244°.
4-p-*Tolylsulphonyl*: m.p. 199–200°.
4-*Benzyl*: m.p. 59°. *B,HCl*: m.p. 228°.

Prelog, Blazěk, *Chem. Abstracts*, 1935, **29**, 2959.
Prelog, Driza, *Chem. Abstracts*, 1934, **28**, 1347.
Pollard, MacDowell, *J. Am. Chem. Soc.*, 1934, **56**, 2199.
Stewart *et al.*, *J. Org. Chem.*, 1948, **13**, 134.

2-Phenylpiperazine.

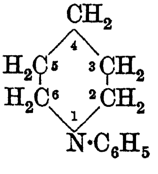
Cryst. from hexane. M.p. 87·5–87·8°. B.p. 138°/10 mm.

B,HCl: cryst. from EtOH.Aq. M.p. 335° decomp.

Diacetyl: cryst. from hexane. M.p. 70–1°.
Dinitroso: cryst. from MeOH. M.p. 70°.
Picrate: m.p. 276° decomp.

Kitchen, Pollard, *J. Am. Chem. Soc.*, 1947, **69**, 854.

N-Phenylpiperidine (1-*Phenylpiperidine*, *pentamethylene-aniline*)



$C_{11}H_{15}N$ MW, 161

B.p. 245–50° (257–8°/752 mm.). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

B,HBr: leaflets from EtOH–Et_2O. M.p. 235°.

B_2,H_2PtCl_6,$2H_2O$: leaflets or needles. Decomp. at 190°.

Picrate: yellow cryst. from EtOH. M.p. 148° (140–2°).

Picrolonate: m.p. 183°.

Methobromide: cryst. from EtOH–Et_2O. M.p. 170° ⟶ *N*-phenylpiperidine + methyl bromide. Somewhat hygroscopic.

Methiodide: cryst. from EtOH–Et_2O. M.p. 146°. Sol. H_2O, EtOH. Gradually turns yellow in air.

Le Fèvre, *J. Chem. Soc.*, 1932, 1378.
Paul, *Bull. soc. chim.*, 1933, **53**, 1489.
v. Braun, *Ber.*, 1907, **40**, 3920.
Horning, Bergstrom, *J. Am. Chem. Soc.*, 1945, **67**, 2110.
Bradley, *J. Chem. Soc.*, 1938, 458.

2-Phenylpiperidine.

Cryst. + $1H_2O$. M.p. 60–1°. The anhyd. base is an oil, b.p. 255–255·5°/767 mm., 110–12°/9 mm. Volatile in steam.

B,HCl: needles from EtOH–Et_2O. M.p. 196–7°.

B,$HAuCl_4$: yellow cryst. M.p. 159–60°.

B_2,H_2PtCl_6: yellow plates. Decomp. about 197°.

Picrate: cryst. from H_2O. M.p. 115–17°.

N-*Benzoyl*: *B,HCl*, m.p. 186–7°.

Adkins, Kuick, Farlow, Wojcik, *J. Am. Chem. Soc.*, 1934, **56**, 2427.
Gabriel, *Ber.*, 1908, **41**, 2013.

3-Phenylpiperidine.

M.p. 14–15°. B.p. 255–6°/740 mm., 139–42°/19 mm. D_{25}^{25} 1·0040. n_D^{25} 1·5473.

B,HCl: m.p. 146–7°.

N-*Benzoyl*: *B,HCl*, m.p. 180–1°.

Walters, McElvain, *J. Am. Chem. Soc.*, 1933, **55**, 4625.
Koelsch, *J. Am. Chem. Soc.*, 1943, **65**, 2093.

4-Phenylpiperidine.

M.p. 57·5–58° (50°). B.p. 255–7°/727 mm. Insol. H_2O. Absorbs CO_2 from the air.

B,HCl: m.p. 186–189·5°.

B,HNO_3: plates from H_2O. M.p. 139°.

Chloroplatinate: orange leaflets. M.p. 204–7°.

N-*Benzoyl*: *hydrochloride*, m.p. 174–5°.

Forsyth, Pyman, *J. Chem. Soc.*, 1930, 401.
Adkins, Kuick, Farlow, Wojcik, *J. Am. Chem. Soc.*, 1934, **56**, 2427.
Bally, *Ber.*, 1887, **20**, 2590.

N-Phenylpiperidine-4-carboxylic Acid.

See N-Phenylhexahydroisonicotinic Acid.

Phenylpiperidylmethane.

See Benzylpiperidine.

Phenylpivalic Acid.

See α: α-Dimethylhydrocinnamic Acid.

Phenylpropandiol.

See Phenylpropylene Glycol *and* Phenyltrimethylene Glycol.

Phenylpropane.

See Cumene *and* Propylbenzene.

Phenylpropanolone.

See 3-Phenylhydroxyacetone.

Phenylpropargyl Alcohol (*Phenylacetylenylcarbinol, phenylpropiolic alcohol, hydroxymethylphenylacetylene*)

$C_6H_5{\cdot}C{\vdots}C{\cdot}CH_2OH$

C_9H_8O MW, 132

B.p. 137–8°/15 mm. (140°/12 mm.). D_{18}^{18} 1·07. n_D^{18} 1·5873. Heat of comb. C_v 1138·1 Cal. Reduces $NH_3.AgNO_3$.

Me ether: $C_{10}H_{10}O$. MW, 146. B.p. 226°, 115°/16 mm. D_4^{20} 1·0016. n_D^{20} 1·5502.

Propyl ether: $C_{12}H_{14}O$. MW, 174. B.p. 137°/17 mm. D_4^{20} 0·9668. n_D^{20} 1·5326.

Acetyl: b.p. 145–7°/16 mm.

Guest, *J. Am. Chem. Soc.*, 1925, **47**, 862.
Bert, *Compt. rend.*, 1930, **191**, 493.
Lai, *Bull. soc. chim.*, 1933, **53**, 682.
Moureu, André, *Ann. chim.*, 1914, 1, 119.

Phenylpropargyl Aldehyde.

See Phenylpropiolic Aldehyde.

Phenylpropiolic Acid

$C_6H_5{\cdot}C{\vdots}C{\cdot}COOH$

$C_9H_6O_2$ MW, 146

Needles from H_2O or CS_2. M.p. 137°. Melts under H_2O at approx. 80°. Very sol. EtOH, Et_2O. $k = 5{\cdot}9 \times 10^{-3}$ at 25°. Heat of comb. C_v 1022·0 Cal. Zn + AcOH ⟶ cinnamic acid. NaHg ⟶ hydrocinnamic acid. Dissolving in conc. H_2SO_4 ⟶ benzoylacetic acid. Hot baryta ⟶ phenylacetylene.

Me ester: $C_{10}H_8O_2$. MW, 160. Cryst. M.p. 26°. B.p. 158–9°/48 mm., 132–3°/16 mm. D_4^{25} 1·0830. n_D^{25} 1·5618. Heat of comb. C_v 7478 cal./gm.

Et ester: $C_{11}H_{10}O_2$. MW, 174. Oil. B.p. 260–70° (rapid dist.), 152–3°/21 mm., 144°/13 mm. D_4^{25} 1·0550. n_D^{25} 1·5502. Heat of comb. C_v 7693 cal./gm.

d-*Amyl ester*: $C_{14}H_{16}O_2$. MW, 216. B.p. 210°/55 mm. D_4^{20} 1·0035. $[\alpha]_D$ + 5·58°.

l-*Menthyl ester*: needles. M.p. 63–4°. B.p. 235–8°/30 mm. D_4^{33} 1·0595. n_D^{17} 1·5239. $[\alpha]_D^{21}$ − 58·65° in $CHCl_3$, $[\alpha]_D^{20}$ − 72·6° in C_6H_6.

d-*Bornyl ester*: yellowish cryst. M.p. 45°. B.p. 228–30°/21 mm. D_4^{15} 1·0884. n_D^{17} 1·55. $[\alpha]_D^{21}$ + 31·05° in $CHCl_3$.

p-*Nitrobenzyl ester*: m.p. 83°.

Chloride: C_9H_5OCl. MW, 164·5. B.p. 115–16°/17 mm. (119°/12 mm.).

Amide: C_9H_7ON. MW, 145. Needles from H_2O. M.p. 108–9° (99–100°). Sol. MeOH, EtOH, $CHCl_3$. Spar. sol. Et_2O, cold H_2O.

Conc. H_2SO_4 ⟶ benzoylacetamide. Hydrazine hydrate in EtOH ⟶ 3-phenylpyrazolone-5.

Nitrile: C_9H_5N. MW, 127. M.p. 41°. B.p. 228–9°, 105–6°/13 mm. $D_4^{41\cdot5}$ 1·0046. $n_D^{41\cdot5}$ 1·58535.

Hydrazide: cryst. from EtOH. M.p. 114° ⟶ 3-phenylpyrazolone-5. *B,HCl*: hygroscopic cryst. M.p. 138–9°. Sol. EtOH, Et_2O. *Picrate*: yellow plates + $1H_2O$. M.p. 105–10°.

Azide: needles from Et_2O. M.p. 55°. Insol. H_2O.

Anilide: needles from EtOH. M.p. 126°.

p-*Toluidide*: plates from EtOH. M.p. 142°.

Abbott, *Organic Syntheses*, 1932, XII, 60.
Curtius, Kenngott, *J. prakt. Chem.*, 1926, **112**, 314.
Bogert, Marcus, *J. Am. Chem. Soc.*, 1919, **41**, 88 (*Note* 1).
Michael, *Ber.*, 1901, **34**, 3648.
Moureu *et al.*, *Ann. chim.*, 1914, **2**, 276.
Reimer, *J. Am. Chem. Soc.*, 1942, **64**, 2510.
Gilman, Haubein, *J. Am. Chem. Soc.*, 1945, **67**, 1420.
Gault, Suprin, *Compt. rend.*, 1946, **222**, 86.

Phenylpropiolic Alcohol.
See Phenylpropargyl Alcohol.

Phenylpropiolic Aldehyde (*Phenylpropargyl aldehyde*)

$C_6H_5{\cdot}C{:}C{\cdot}CHO$

C_9H_6O MW, 130

Oil with odour resembling cinnamaldehyde. B.p. 127–8°/28 mm., 118°/17 mm., 112–13°/15 mm., 104–5°/11 mm. $D_4^{12\cdot6}$ 1·0680. $n_D^{12\cdot6}$ 1·60785. Decomp. on dist. at atmospheric press. Heat of comb. C_v 1081·7 Cal. Aq. alkalis ⟶ phenylacetylene + H·COOH.

Oxime: needles from ligroin. M.p. 108°. Acetic anhydride ⟶ phenylpropiolic nitrile.

Di-Et acetal: b.p. 144–5°/14 mm. D^{13} 0·9940. n_D^{13} 1·52216.

Kalff, *Rec. trav. chim.*, 1927, **46**, 594.
Auwers, Seyfried, *Ann.*, 1930, **484**, 224.
Moureu, Delange, *Bull. soc. chim.*, 1904, **31**, 1329.

1-Phenylpropionaldehyde.
See Hydratropic Aldehyde.

2-Phenylpropionaldehyde.
See Hydrocinnamaldehyde.

1-Phenylpropionic Acid.
See Hydratropic Acid.

2-Phenylpropionic Acid.
See Hydrocinnamic Acid.

β-Phenylpropiophenone.
See α-Methyldeoxybenzoin.

1-Phenylpropyl Alcohol.
See Ethylphenylcarbinol.

2-Phenylpropyl Alcohol (*β-Hydroxyisopropylbenzene, β-hydroxycumene, hydratropic alcohol*)

$$CH_3{\cdot}\underset{}{\overset{C_6H_5}{CH}}{\cdot}CH_2OH$$

$C_9H_{12}O$ MW, 136

dl-.

B.p. 114°/14 mm. D^0 1·017.

Acid phthalate: prisms from CS_2. M.p. 79°.

Acetyl: b.p. 125°/14 mm., 103–5°/10 mm.

Benzoyl: b.p. 198–200°/20 mm.

p-*Nitrobenzoyl*: plates. M.p. 65°.

Phenylurethane: needles. M.p. 156°.

Ramart, Amagat, *Ann. chim.*, 1927, **8**, 263.
Cohen, Marshall, Woodman, *J. Chem. Soc.*, 1915, **107**, 897.

3-Phenylpropyl Alcohol.
See Hydrocinnamyl Alcohol.

1-Phenylpropylamine (*α-Aminopropylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH(NH_2){\cdot}C_6H_5$

$C_9H_{13}N$ MW, 135

Oil. B.p. 204–6°/748 mm., 100–5°/35 mm., 99–100°/16 mm. D_0^0 0·9560, D_0^{25} 0·9347. n^{25} 1·51726. Spar. sol. H_2O. Absorbs CO_2.

B,HCl: needles from EtOH. M.p. 189·5° (194°).

N-*Benzoyl*: needles from EtOH. M.p. 115–16°.

N-*Benzenesulphonyl*: needles from EtOH. M.p. 81°.

N-*Me*: α-methylaminopropylbenzene. $C_{10}H_{15}N$. MW, 149. B.p. 96°/20 mm. *B,HCl*: needles or prisms. M.p. 153°.

N-*Et*: α-ethylaminopropylbenzene. $C_{11}H_{17}N$. MW, 163. B.p. 207–8°/729 mm., 99°/20 mm. *B,HCl*: needles or prisms. M.p. 180°.

N-*Phenyl*: α-phenylpropylaniline, α-anilinopropylbenzene. $C_{15}H_{17}N$. MW, 211. Viscous oil. B.p. 192°/20 mm. *B,HCl*: needles from EtOH–Et_2O. M.p. 187°. *B,HNO₃*: needles or prisms. M.p. 174°. Part. hyd. by H_2O.

N-*Benzyl*: *see* α-Ethyldibenzylamine.

d-.

Oil. B.p. 204–6°, $[\alpha]_D^{17}$ + 20·15° (+ 9·5°, + 4·57°).

l-.

Oil. $[\alpha]_D^{17}$ − 19·85° (− 10·5°).

Hartung, Munch, *J. Am. Chem. Soc.*, 1931, **53**, 1878.
Konowalow, *Chem. Zentr.*, 1894, I, 465.
Busch, Leefhelm, *J. prakt. Chem.*, 1908, **77**, 8.
Levine, Rothin, Kuna, *J. Biol. Chem.*, 1937, **120**, 777.
Little, M'Lean, Wilson, *J. Chem. Soc.*, 1940, 336.
Crossley, Moore, *J. Org. Chem.*, 1944, **9**, 529.

2-Phenylpropylamine (*β-Aminoisopropylbenzene, β-aminocumene*)

$$CH_3\cdot CH(C_6H_5)\cdot CH_2NH_2$$

$C_9H_{13}N$ MW, 135

Oil with fishy odour. B.p. 210°, 104°/21 mm. D_4^{20} 0·9433. n_D^{20} 1·5255. Misc. with EtOH, Et_2O, C_6H_6. Very spar. sol. H_2O. Absorbs H_2O and CO_2 from the air.

B,HCl: m.p. 123–4°.
B_2,H_2PtCl_6: m.p. 229° decomp.
B,HAuCl$_4$: m.p. 124°.
N-*Me*: liq. B.p. 66–9°/1 mm. n_D^{25} 1·5146.
N-*Benzoyl*: cryst. from EtOH. M.p. 85°.
Picrate: m.p. 182°.

v. Braun, Grabowski, Kirschbaum, *Ber.*, 1913, **46**, 1281.
Freund, König, *Ber.*, 1893, **26**, 2875.
Hartung, Munch, *J. Am. Chem. Soc.*, 1931, **53**, 1878.
Weston, Ruddy, Suter, *J. Am. Chem. Soc.*, 1943, **65**, 674.
Suter, Weston, U.S.P. 2,441,518, (*Chem. Abstracts*, 1948, **42**, 5469).
Crossley, Moore, *J. Org. Chem.*, 1944, **9**, 529.

3-Phenylpropylamine (*γ-Aminopropylbenzene*)

$$C_6H_5\cdot CH_2\cdot CH_2\cdot CH_2NH_2$$

$C_9H_{13}N$ MW, 135

B.p. 221·5°/755 mm., 75–80°/1 mm. D_4^{25} 0·9760. Misc. with EtOH, Et_2O. Mod. sol. H_2O with strongly alk. reaction. Absorbs CO_2.

B,HCl: leaflets from EtOH–Et_2O. M.p. 218°.
B_2,H_2PtCl_6: yellow leaflets from H_2O. Decomp. at 233°.
Neutral oxalate: needles from H_2O. M.p. 190° decomp.
Acid oxalate: prisms from EtOH. M.p. 156°. Very sol. hot H_2O.
Picrate: lemon-yellow needles from H_2O. M.p. 152–3°.
N-*Me*: γ-methylaminopropylbenzene. $C_{10}H_{15}N$. MW, 149. Odourless oil. B.p. 116°/20 mm. (133–5°/18 mm.). *B_2,H_2PtCl_6*: red leaflets from H_2O. M.p. 188°. *Picrate*: needles from EtOH. M.p. 93–4°.
N-*Di-Me*: γ-dimethylaminopropylbenzene. $C_{11}H_{17}N$. MW, 163. B.p. 222–4°, 117–18°/26 mm., 99°/14 mm. *B,HCl*: m.p. 146°. *B_2,H_2PtCl_6*: orange cryst. M.p. 152°. *Methobromide*: cryst. from EtOH–Et_2O. M.p. 143°. *Methiodide*: needles from EtOH. M.p. 179°.
N-*Et*: γ-ethylaminopropylbenzene. $C_{11}H_{17}N$. MW, 163. B.p. 124–6°/25 mm., 118°/16 mm. *B_2,H_2PtCl_6*: needles. M.p. 134–5°.
N-*Di-Et*: γ-diethylaminopropylbenzene. $C_{13}H_{21}N$. MW, 191. B.p. 137–9°/22 mm.
N-*Propyl*: γ-propylaminopropylbenzene. $C_{12}H_{19}N$. MW, 177. B.p. 134°/17 mm. *Picrate*: orange cryst. from EtOH–Et_2O. M.p. 97°.
N-*Dipropyl*: γ-dipropylaminopropylbenzene. $C_{15}H_{25}N$. MW, 219. B.p. 158–60°/17 mm. *B_2,H_2PtCl_6*: red cryst. M.p. 91–3°.
N-*Benzoyl*: cryst. from H_2O. M.p. 57–8°.

Hartung, Munch, *J. Am. Chem. Soc.*, 1931, **53**, 1878.
Goodyear, F.P., 751,712, (*Chem. Zentr.*, 1934, I, 126).
v. Braun, Aust, *Ber.*, 1916, **49**, 504.
Emde, *Ann.*, 1912, **391**, 93.
v. Braun, *Ber.*, 1910, **43**, 3218.
Gabriel, Eschenbach, *Ber.*, 1897, **30**, 1128.
Michaelis, Jacobi, *Ber.*, 1893, **26**, 2160.
Kindler, Hedemann, Scharfe, *Ann.*, 1948, **560**, 215.

α-Phenylpropylaniline.
See under 1-Phenylpropylamine.

2-Phenylpropyl chloride.
See β-Chlorocumene.

Phenylpropylene.
See Allylbenzene *and* α-, and β-Methylstyrenes.

1-Phenylpropylene Glycol (*αβ-Dihydroxypropylbenzene, α-methyl-α′-phenylethylene glycol, 1-phenylpropandiol-1 : 2*)

$$CH_3\cdot CH(OH)\cdot CH(OH)\cdot C_6H_5$$

$C_9H_{12}O_2$ MW, 152

Exists in two forms:
(i) Plates from Et_2O–pet. ether. M.p. 56–7° (52–3°). Very sol. H_2O, EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Ox. ⟶ benzaldehyde and acetaldehyde.
Dibenzoyl: needles. M.p. 101°.
(ii) Plates from Et_2O. M.p. 92–3°. 20% H_2SO_4 ⟶ phenylacetone. Ox. ⟶ benzaldehyde and acetaldehyde.
Dibenzoyl: m.p. 76–7°.

Lévy, Dvoleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1765.
Zincke, Zahn, *Ber.*, 1910, **43**, 851.
Tiffeneau, *Compt. rend.*, 1906, **142**, 1538.
Fourneau, Benoit, *Bull. soc. chim.*, 1945, **12**, 985.

2-Phenylpropylene Glycol (*αβ-Dihydroxyisopropylbenzene, α-methyl-α-phenylethylene glycol, 2-phenylpropandiol-1 : 2*)

$$CH_3\cdot C(C_6H_5)(OH)\cdot CH_2OH$$

$C_9H_{12}O_2$ MW, 152

Needles from pet. ether or ligroin–Et_2O. M.p. 44·5° (40–1°). B.p. 158–60°/25 mm., 153°/16 mm. Sol. H_2O, EtOH, Et_2O. Spar. sol. ligroin.

Danilow, Venus-Danilowa, *Ber.*, 1927, **60**, 1063.
Stoermer, *Ber.*, 1906, **39**, 2297.

3-Phenylpropylene Glycol (*βγ-Dihydroxypropylbenzene*, *3-phenylpropandiol*-1 : 2, *benzylethylene glycol*)

$$C_6H_5{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2OH$$

$C_9H_{12}O_2$ MW, 152

B.p. 163–5°/15 mm.

Diacetyl: b.p. 282–6°, 163–6°/13 mm. D^0 1·128.

Dibenzoyl: m.p. 74–5°.

1-*Phenyl ether*: $C_{15}H_{16}O_2$. MW, 228. Plates from C_6H_6. M.p. 91–2°. Insol. H_2O.

Hershberg, *Helv. Chim. Acta*, 1934, **17**, 351

Fourneau, *Chem. Zentr.*, 1910, I, 1134.

α-Phenylpropyl-malonic Acid (2-*Phenylbutane*-1 : 1-*dicarboxylic acid*, *ethylphenylisosuccinic acid*)

$$CH_3{\cdot}CH_2{\cdot}\overset{C_6H_5}{CH}{\cdot}CH{<}\begin{smallmatrix}COOH\\COOH\end{smallmatrix}$$

$C_{12}H_{14}O_4$ MW, 222

Plates + $1H_2O$ from H_2O. M.p. 74°. Sol. EtOH, Et_2O, Me_2CO. At 150° ⟶ 2-phenylvaleric acid.

Di-Et ester: $C_{16}H_{22}O_4$. MW, 278. B.p. 187–8°/22 mm.

Reynolds, *Am. Chem. J.*, 1910, **44**, 315.

γ-Phenylpropyl-malonic Acid (4-*Phenylbutane*-1 : 1-*dicarboxylic acid*)

$$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH{<}\begin{smallmatrix}COOH\\COOH\end{smallmatrix}$$

$C_{12}H_{14}O_4$ MW, 222

Cryst. from C_6H_6–ligroin. M.p. 98° (94°). Sol. most org. solvents except ligroin. Heated above m.p. or better at 200° under reduced press. ⟶ 4-phenylvaleric acid.

Di-Me ester: $C_{14}H_{18}O_4$. MW, 250. B.p. 183–4°/10 mm.

Di-Et ester: b.p. 189–94°/13 mm.

Et ester-nitrile: $C_{14}H_{17}O_2N$. MW, 231. Oil. B.p. 192–3°/11 mm.

v. Braun, Kruber, *Ber.*, 1912, **45**, 386.

Przewalski, *Chem. Zentr.*, 1923, III, 665.

Borsche, *Ber.*, 1912, **45**, 622.

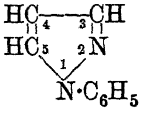
1-Phenylpropyne.

See Methylphenylacetylene.

1-Phenylpropynol-1.

See Ethynylphenylcarbinol.

1-Phenylpyrazole



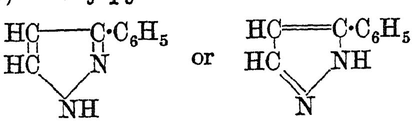
$C_9H_8N_2$ MW, 144

Needles. M.p. 11–11·5°. B.p. 246–7°. Sol. EtOH, Et_2O. Spar. sol. warm H_2O.

Chloroaurate: yellow needles from EtOH–HCl. M.p. 181°.

Alvisi, *Gazz. chim. ital.*, 1892, **22**, i, 161.

3(5)-Phenylpyrazole



$C_9H_8N_2$ MW, 144

Needles from H_2O. M.p. 79°. B.p. 313–14°, 177–8°/11 mm. Sol. EtOH, Et_2O. $D_4^{99·8}$ 1·0818. $n_{He}^{100·8}$ 1·58890.

B,HCl: cryst. powder. M.p. 144–5°.

B,HNO₃: needles from Et_2O. M.p. 126° decomp. Sol. H_2O, EtOH. Spar. sol. Et_2O, Me_2CO, C_6H_6.

N-*Acetyl*: needles from ligroin. M.p. 64–5°. B.p. 157–8°/10 mm. Sol. Me_2CO, C_6H_6. Spar. sol. EtOH, ligroin. $D_4^{100·3}$ 1·0778. $n_{He}^{100·3}$ 1·56064.

N-o-*Nitrobenzoyl of 3-phenylpyrazole*: cryst. from MeOH. M.p. 107·5–108·5°. Sol. Me_2CO, C_6H_6. Spar. sol. MeOH.

N-o-*Nitrobenzoyl of 5-phenyl pyrazole*: needles from MeOH. M.p. 151–2°. Sol. Me_2CO. Spar. sol. MeOH, C_6H_6.

Picrate: m.p. 168–70°.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 89.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 194.

Auwers, Schmidt, *Ber.*, 1925, **58**, 538.

Buchner, Hachumian, *Ber.*, 1902, **35**, 38.

Cusmano, *Gazz. chim. ital.*, 1940, **70**, 227.

Bowden, Jones, *J. Chem. Soc.*, 1946, 953.

For non-equivalence of positions 3 and 5 see Auwers, *Ann.*, 1933, **508**, 57.

4-Phenylpyrazole.

Plates from EtOH. M.p. 230°. Sol. EtOH, C_6H_6. Spar. sol. Et_2O.

B,HCl: needles. M.p. 215–18°.

N-*Acetyl*: yellow needles from EtOH. M.p. 81·5–82·5°. B.p. 159–61°. Sol. Me_2CO, C_6H_6. Spar. sol. ligroin.

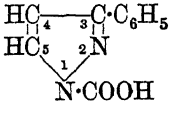
Picrate: yellow cryst. from EtOH. M.p. 155°.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 194.

Rupe, Huber, *Helv. Chim. Acta*, 1927, **10**, 848.

Buchner, Perkel, *Ber.*, 1903, **36**, 3777.

3-Phenylpyrazole-1-carboxylic Acid



$C_{10}H_8O_2N_2$ MW, 188

Et ester: $C_{12}H_{12}O_2N_2$. MW, 216. B.p. 193°/13 mm. D_4^{19} 1·1694. n_{He}^{19} 1·57904.

Amide: $C_{10}H_9ON_3$. MW, 187. Needles from

Me_2CO. M.p. 142–3°. Sol. ord. org. solvents.
Picrate : yellow needles from Et_2O. M.p. 165–6°.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 89.
Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 197.
Auwers, Ottens, *Ber.*, 1925, **58**, 2076.

4-Phenylpyrazole-1-carboxylic Acid.

Et ester : plates from ligroin. M.p. 78–78·5°. B.p. 192°/9 mm. Very sol. ord. org. solvents. $D_4^{99.9}$ 1·1055. $n_{He}^{99.5}$ 1·54637.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 193.

5-Phenylpyrazole-1-carboxylic Acid.

Me ester : $C_{11}H_{10}O_2N_2$. MW, 202. Needles from EtOH. M.p. 152°. Sol. ord. org. solvents.
Et ester : cryst. from C_6H_6. M.p. 58–9°. Very sol. MeOH, EtOH. Spar. sol. C_6H_6. $D_4^{78.4}$ 1·1165. $n_{He}^{78.4}$ 1·53860.
Amide : plates from C_6H_6. M.p. 133–4°. Very sol. AcOH. Mod. sol. EtOH, Me_2CO, C_6H_6. Spar. sol. Et_2O.

Auwers, Breyhan, *J. prakt. Chem.*, 1935, **143**, 267.
Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 89.
Auwers, Ottens, *Ber.*, 1925, **58**, 2077.

1-Phenylpyrazole-3-carboxylic Acid.

Needles from H_2O. M.p. 146°. Sol. EtOH, Et_2O. Spar. sol. cold H_2O.
Me ester : $C_{11}H_{10}O_2N_2$. MW, 202. Needles. M.p. 77°.

Claisen, Roosen, *Ann.*, 1894, **278**, 294.

4-Phenylpyrazole-3-carboxylic Acid.

Plates. M.p. 252–3° with decomp. to 4-phenylpyrazole. Spar. sol. hot H_2O, EtOH, Me_2CO. Insol. $CHCl_3$.
Me ester : prisms from MeOH. M.p. 188–90°. N-*Acetyl* : needles from EtOH. M.p. 129·5–130·5°. Sol. Et_2O. Mod. sol. EtOH. Spar. sol. ligroin.
Et ester : $C_{12}H_{12}O_2N_2$. MW, 216. Plates from EtOH. M.p. 164–5°. Sol. MeOH. Spar. sol. Et_2O.
Nitrile : needles from C_6H_6. M.p. 149·5–150°. Very sol. EtOH, Et_2O. Mod. sol. C_6H_6. Spar. sol. H_2O, ligroin.

Auwers, Ungemach, *Ber.*, 1933, **66**, 1204, 1207.
Auwers, Cauer, *Ann.*, 1929, **470**, 302.
Kohler, Steele, *J. Am. Chem. Soc.*, 1919, **41**, 1104.

5(3)-Phenylpyrazole-3(5)-carboxylic Acid.

Needles from H_2O or EtOH. M.p. 234° decomp. Sol. MeOH, EtOH. Insol. C_6H_6.
Me ester : $C_{11}H_{10}O_2N_2$. MW, 202. Prisms from MeOH or Et_2O. M.p. 182°.

Et ester : $C_{12}H_{12}O_2N_2$. MW, 216. Needles from MeOH or Et_2O. M.p. 140°. Sol. hot H_2O, EtOH, Et_2O. Very sol. $CHCl_3$. N-*Acetyl* : needles from C_6H_6. M.p. 109–10°.
Hydrazide : m.p. 205°.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 93.
Auwers, Mausolf, *Ber.*, 1927, **60**, 1732.
Bülow, *Ber.*, 1904, **37**, 2201.
Cusmano, *Gazz. chim. ital.*, 1940, **70**, 227.

1-Phenylpyrazole-4-carboxylic Acid.

Needles from H_2O. M.p. 219–20°. Sol. EtOH. Spar. sol. H_2O, Et_2O. Sublimes in needles.
Me ester : needles from MeOH. M.p. 128–9°.
Et ester : prisms from EtOH. M.p. 96–7°.

Wislicenus, Bindemann, *Ann.*, 1901, **316**, 36.

5-Phenylpyrazole-4-carboxylic Acid.

Needles from H_2O. M.p. 260°. Sol. EtOH. Spar. sol. H_2O. Insol. Et_2O.
Me ester : needles from ligroin. M.p. 111·5–112·5°.
Et ester : needles from EtOH.Aq. M.p. 85–6°.
Anilide : cryst. + 1EtOH from EtOH. M.p. 182°.
o-*Anisidide* : needles from EtOH.Aq. M.p. 127°. Sol. most org. solvents.
p-*Anisidide* : needles from EtOH.Aq. M.p. 161°.

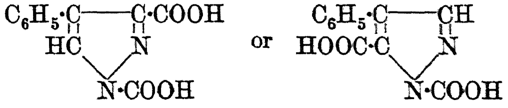
Auwers, Ungemach, *Ber.*, 1933, **66**, 1207.
Dains, Long, *J. Am. Chem. Soc.*, 1921, **43**, 1201.

1-Phenylpyrazole-5-carboxylic Acid.

Needles from H_2O. M.p. 183°. Sol. EtOH, Et_2O. Spar. sol. cold H_2O.
Me ester : $C_{11}H_{10}O_2N_2$. MW, 202. Needles. M.p. 67°.
Chloride : needles from ligroin. M.p. 53°. B.p. 152°/15 mm.

Rojahn, Seitz, *Ann.*, 1924, **437**, 303.
Claisen, Roosen, *Ann.*, 1894, **278**, 292.
Cusmano, *Gazz. chim. ital.*, 1940, **70**, 227.
Panizzi, Monti, *Gazz. chim. ital.*, 1947, **77**, 556.

4-Phenylpyrazole-1 : 3(5)-dicarboxylic Acid

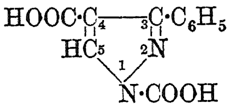


$C_{11}H_8O_4N_2$ MW, 232

3(5)-*Et ester* : $C_{13}H_{12}O_4N_2$. MW, 260. *Anilide* : needles from ligroin. M.p. 105–6°. Sol. ord. org. solvents.
1-*Me*-3(5)-*Et ester* : $C_{14}H_{14}O_4N_2$. MW, 274. Needles from EtOH. M.p. 75–6°. Sol. ord. org. solvents.

Auwers, Ungemach, *Ber.*, 1933, **66**, 1203.

3 - Phenylpyrazole - 1 : 4 - dicarboxylic Acid



$C_{11}H_8O_4N_2$ MW, 232

4-*Et ester*: $C_{13}H_{12}O_4N_2$. MW, 260. Cryst. from EtOH. M.p. 85–6°. *Anilide*: cryst. from ligroin. M.p. 136–7°. Sol. ord. org. solvents.

Di-Et ester: $C_{15}H_{16}O_4N_2$. MW, 288. Needles from ligroin. M.p. 57·5–58·5°. Sol. ord. org. solvents. Spar. sol. ligroin.

Auwers, Ungemach, *Ber.*, 1933, **66**, 1207.

3(5)-Phenylpyrazole-1 : 5(3)-dicarboxylic Acid.

1-*Me*-5(3)-*Et ester*: $C_{14}H_{14}O_4N_2$. MW, 274. Needles from ligroin. M.p. 80–1°. Sol. ord. org. solvents.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 93.

1 - Phenylpyrazole - 3 : 4 - dicarboxylic Acid.

Plates from H_2O. M.p. 234° decomp. Spar. sol. cold H_2O, Et_2O.

Di-Me ester: $C_{13}H_{12}O_4N_2$. MW, 260. Tablets. M.p. 97–8°.

Balbiano, *Gazz. chim. ital.*, 1898, **28**, i, 385.

5 - Phenylpyrazole - 3 : 4 - dicarboxylic Acid.

Cryst. M.p. 235° decomp. Very sol. hot H_2O, EtOH. Mod. sol. Et_2O. Resorcinol ⟶ fluorescein reaction.

Buchner, *Ber.*, 1894, **27**, 3247.
Sjollema, *Ann.*, 1894, **279**, 252.

1 - Phenylpyrazole - 3 : 5 - dicarboxylic Acid.

Plates from EtOH. M.p. 270–2° decomp. to 1-phenylpyrazole-3-carboxylic acid. Sol. Et_2O. Spar. sol. cold H_2O, $CHCl_3$, ligroin, C_6H_6.

Di-NH_4 salt: plates. M.p. 210–15° decomp. Very sol. H_2O.

Di-Me ester: $C_{13}H_{12}O_4N_2$. MW, 260. M.p. 124–5°.

Diamide: $C_{11}H_{10}O_2N_4$. MW, 230. Plates from EtOH. M.p. 190°. Very sol. EtOH.

Claisen, Roosen, *Ann.*, 1894, **278**, 286.
Tracy, Elderfield, *J. Org. Chem.*, 1941, **6**, 70.

4 - Phenylpyrazole - 3 : 5 - dicarboxylic Acid.

Needles + $2H_2O$ from H_2O. Loses H_2O at 130°. M.p. 243–6°. Dist. ⟶ 4-phenylpyrazole.

Me-Et ester: $C_{14}H_{14}O_4N_2$. MW, 274. Rhombohedra or needles from Et_2O. M.p. 104–5°.

Di-Et ester: $C_{15}H_{16}O_4N_2$. MW, 288. Needles from EtOH.Aq. M.p. 96°.

Behaghel, Buchner, *Ber.*, 1902, **35**, 34.

1 - Phenylpyrazole - 4 : 5 - dicarboxylic Acid.

Prisms and plates from H_2O. M.p. 216°. Sol. EtOH. Very sol. hot H_2O. Spar. sol. cold H_2O, Et_2O, $CHCl_3$. Dist. ⟶ 1-phenylpyrazole-4-carboxylic acid. Resorcinol ⟶ greenish-red fluorescence.

Ba salt: needles from H_2O. Spar. sol. cold H_2O.

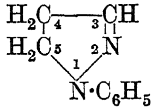
Di-Me ester: $C_{13}H_{12}O_4N_2$. MW, 260. Needles from MeOH. M.p. 75–6°.

Diamide: $C_{11}H_{10}O_2N_4$. MW, 230. Needles from H_2O. M.p. 253–5°.

Dianilide: plates. M.p. 205–6°. Sol. EtOH, AcOEt. Spar. sol. Et_2O, ligroin.

Claisen, *Ann.*, 1897, **295**, 315.

1-Phenylpyrazoline



$C_9H_{10}N_2$ MW, 146

Plates from ligroin. M.p. 51–2°. B.p. 273–4°/754 mm. Sol. hot H_2O, EtOH, Et_2O, C_6H_6. Slightly basic. Volatile in steam from slightly acid sol.

Michaelis, Lampe, *Ann.*, 1893, **274**, 319.

3-Phenylpyrazoline.

M.p. 44–5°. B.p. 164°/17 mm. Spar. sol. pet. ether.

Picrate: cryst. from MeOH. M.p. 142–3°.

Nitroso deriv.: m.p. 152·5–153·5°.

Auwers, Heimke, *Ann.*, 1927, **458**, 207.

4-Phenylpyrazoline.

Oil. Oxidises in air to 4-phenylpyrazole.

B,HCl: cryst. from EtOH. M.p. 162°. Sol. H_2O, EtOH. Insol. Et_2O.

$B_2,(COOH)_2$: cryst. from EtOH. M.p. 120°.

B_2,H_2PtCl_6: m.p. 116–17°.

Oliveri, Mandala, *Gazz. chim. ital.*, 1910, **40**, i, 117.
Buchner, Dessauer, *Ber.*, 1903, **36**, 3777.

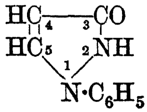
5-Phenylpyrazoline.

Colourless oil. B.p. 153°/15 mm. $D_4^{15\cdot8}$ 1·1068.

Picrate: m.p. 117–18°.

Auwers, Heimke, *Ann.*, 1927, **458**, 208.

1-Phenylpyrazolone-3



$C_9H_8ON_2$ MW, 160

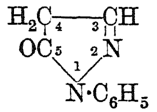
Needles from H_2O. M.p. 154°. Sol. hot EtOH, $CHCl_3$, C_6H_6, conc. HCl, alkalis. Spar. sol. hot H_2O.

B,HCl: needles. M.p. 111°.

N-*Acetyl*: needles from ligroin. M.p. 62–3°.

Harries, Loth, *Ber.*, 1896, **29**, 519.

1-Phenylpyrazolone-5



$C_9H_8ON_2$ MW, 160

Prisms and plates from AcOEt. M.p. 118–19°. Sol. acids, alkalis.

B,HCl: needles. M.p. 165°.

Isonitroso deriv.: orange-yellow needles from AcOH.Aq. M.p. 160° decomp.

Claisen, Haase, *Ber.*, 1895, **28**, 38.

3-Phenylpyrazolone-5.

Plates from EtOH. M.p. 236° decomp. Insol. cold EtOH, Et_2O, C_6H_6.

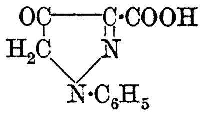
B,HCl: needles. M.p. 196°.

N-*Acetyl*: plates from EtOH. M.p. 127–8°. Spar. sol. cold EtOH, Et_2O, C_6H_6.

Isonitroso deriv.: yellow needles from H_2O. M.p. 188°. Sol. EtOH, Et_2O, alkalis.

Rothenburg, *J. prakt. Chem.*, 1895, **52**, 23.
De, Rekshit, *J. Indian Chem. Soc.*, 1936, **13**, 509, (*Chem. Abstracts*, 1937, **31**, 1403).
Weissberger, Porter, *J. Am. Chem. Soc.*, 1943, **65**, 1495.

1 - Phenyl - 4 - pyrazolone - 3 - carboxylic Acid (1-*Phenyl-isopyrazolone*-3-*carboxylic acid*)



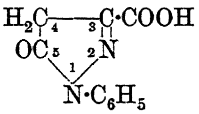
$C_{10}H_8O_3N_2$ MW, 204

Me ester: $C_{11}H_{10}O_3N_2$. MW, 218. Needles from MeOH. M.p. 85–7°. $FeCl_3$ ⟶ blue col.

Et ester: $C_{12}H_{12}O_3N_2$. MW, 232. Pale yellow needles from EtOH. M.p. 258–60°. $FeCl_3$ ⟶ blue col.

Favrel, *Compt. rend.*, 1913, **156**, 1912.

1 - Phenyl - 5 - pyrazolone - 3 - carboxylic Acid



$C_{10}H_8O_3N_2$ MW, 204

Small needles from EtOH or hot H_2O. Loses CO_2 at 230°, m.p. 261°. Sol. EtOH. Insol. H_2O, Et_2O. $FeCl_3$ ⟶ blue col.

Me ester: $C_{11}H_{10}O_3N_2$. MW, 218. Cryst. from MeOH. M.p. 197°.

Et ester: $C_{12}H_{12}O_3N_2$. MW, 232. Prisms from EtOH. M.p. 180–2°. $FeCl_3$ ⟶ blue col.

Et ether: plates from H_2O. M.p. 152–3°. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. cold H_2O.

Et ester: needles from EtOH.Aq. M.p. 83–4°.

Leighton, *J. Am. Chem. Soc.*, 1898, **20**, 679.

1 - Phenyl - 5 - pyrazolone - 4 - carboxylic Acid.

M.p. 91–2° decomp. Sol. EtOH, Me_2CO.

Et ester: needles from EtOH.Aq. M.p. 117–18°. Sol. EtOH, Et_2O, Me_2CO, C_6H_6, alkalis. $FeCl_3$ ⟶ dark red col.

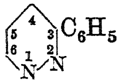
Ruhemann, *J. Chem. Soc.*, 1893, **63**, 878.

2 - Phenyl - 5 - pyrazolone - 4 - carboxylic Acid.

Cryst. from EtOH. M.p. 216° decomp. Dist. ⟶ 2-phenylpyrazolone-5.

Michaelis, Remy, *Ber.*, 1907, **40**, 1020.

3-Phenylpyridazine (3-*Phenyl*-1 : 2-*diazine*)



$C_{10}H_8N_2$ MW, 156

Needles from H_2O or ligroin. M.p. 102–3°. B.p. 330–2°. Sol. EtOH, Et_2O. Sublimes.

B,MeI: yellow needles from MeOH–Et_2O. M.p. 179°. Sol. MeOH.

B,HAuCl₄: yellow needles. M.p. 159°.

Picrate: cryst. from EtOH. M.p. 127°.

Gabriel, Colman, *Ber.*, 1899, **32**, 401.

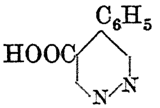
4-Phenylpyridazine (4-*Phenyl*-1 : 2-*diazine*).

Cryst. from ligroin. M.p. 86–86·5°. Sol. EtOH, Et_2O, C_6H_6, acids. Spar. sol. ligroin.

B_2,H_2PtCl_6,H_2O: cryst. M.p. 295–300° decomp. Spar. sol. H_2O, EtOH.

Stoermer, Fincke, *Ber.*, 1909, **42**, 3130.

4-Phenylpyridazine-5-carboxylic Acid

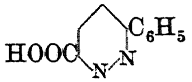


$C_{11}H_8O_2N_2$ MW, 200

Slightly brownish crystals. M.p. 220–1° decomp. Mod. sol. hot EtOH. Spar. sol. hot H_2O. Insol. Et_2O, C_6H_6. Heat with $BaCO_3$ in vacuo ⟶ 4-phenylpyridazine.

Stoermer, Finke, *Ber.*, 1909, **42**, 3130.

3-Phenylpyridazine-6-carboxylic Acid

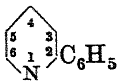


$C_{11}H_8O_2N_2$ MW, 200

Needles from H_2O. M.p. 130–1°.

Paal, Dencks, *Ber.*, 1903, **36**, 494.

2-Phenylpyridine (α-*Pyridylbenzene*)



$C_{11}H_9N$ MW, 155

Oil. B.p. 268–9°. Misc. with EtOH, Et_2O, but not H_2O. Difficultly volatile in steam. Neutral $KMnO_4$ ⟶ benzoic acid. Acid $KMnO_4$ ⟶ picolinic acid.

$B_2,H_2PtCl_6,2H_2O$: orange-yellow needles. M.p. 204°. Insol. H_2O, EtOH, Et_2O.

Picrate: rhombic prisms. M.p. 175°. Very sol. hot EtOH.

Ziegler, Zeiser, *Ber.*, 1930, **63**, 1851.
Bergstrom, McAllister, *J. Am. Chem. Soc.*, 1930, **52**, 2847.
Haworth, Heilbron, Hey, *J. Chem. Soc.*, 1940, 349.
Elks, Hey, *J. Chem. Soc.*, 1943, 441.

3-Phenylpyridine (β-*Pyridylbenzene*).

Pale yellow oil. B.p. 269–70°/749 mm. Immiscible with H_2O. Sol. EtOH, Et_2O, dil. acids. Difficultly volatile in steam. $KMnO_4$ ⟶ nicotinic acid.

Picrate: pale yellow needles from EtOH. M.p. 159–60°.

Skraup, Cobenzl, *Monatsh.*, 1883, **4**, 456.
Haworth, Heilbron, Hey, *J. Chem. Soc.*, 1940, 349.
Elks, Hey, *J. Chem. Soc.*, 1943, 441.

4-Phenylpyridine (γ-*Pyridylbenzene*).

Plates from H_2O. M.p. 77–8°. B.p. 274–5°. Mod. sol. hot H_2O. $KMnO_4$ ⟶ isonicotinic acid. Na + EtOH ⟶ 4-phenylpiperidine.

B,MeI: nearly colourless cryst.

$B_2,H_2Cr_2O_7$: orange needles from H_2O. M.p. 155° decomp.

Picrate: orange needles. M.p. 195–6°.

Overhoff, Tilman, *Rec. trav. chim.*, 1929, **48**, 993.
Haworth, Heilbron, Hey, *J. Chem. Soc.*, 1940, 349.
Elks, Hey, *J. Chem. Soc.*, 1943, 441.

6-Phenylpyridine-2-carboxylic Acid.

See 6-Phenylpicolinic Acid.

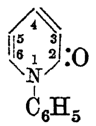
6-Phenylpyridine-3-carboxylic Acid.

See 6-Phenylnicotinic Acid.

Phenylpyridine-dicarboxylic Acid.

See Phenylcinchomeronic Acid, Phenyldinicotinic Acid *and* Phenylquinolinic Acid.

1-Phenyl-α-pyridone



$C_{11}H_9ON$ MW, 171

Cryst. from EtOH or C_6H_6. M.p. 128°. B.p. above 300°.

Tschitschibabin, Geletzki, *Ber.*, 1924, **57**, 1159.

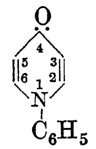
6-Phenyl-α-pyridone.

Flat rods from MeOH. M.p. 195°. Sol. EtOH. Spar. sol. C_6H_6. Insol. H_2O, Et_2O. Zn dust dist. ⟶ 2-phenylpyridine. PCl_3 ⟶ 6-chloro-2-phenylpyridine.

B,HCl: pale yellow needles. M.p. 104° (101–3°).

Leben, *Ber.*, 1896, **29**, 1678.
Allen, Ball, *J. Am. Chem. Soc.*, 1937, **59**, 686.

1-Phenyl-γ-pyridone



$C_{11}H_9ON$ MW, 171

Needles + $2H_2O$ from H_2O, m.p. anhyd. 104–5°: cryst. from C_6H_6, m.p. 116°. Very sol. warm H_2O, EtOH. Spar. sol. Et_2O.

B,MeI: rhomboids. M.p. 146°.

Picrate: yellow leaflets from EtOH. M.p. 190°.

Borsche, Bonacker, *Ber.*, 1921, **54**, 2682.

6-Phenyl-γ-pyridone.

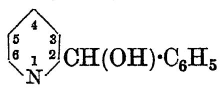
Cryst. + $\frac{1}{2}H_2O$ from H_2O. M.p. 155°.

Borsch, Peter, *Ann.*, 1927, **453**, 158.

Phenyl-2-pyridylamine.

See 2-Anilinopyridine.

Phenyl-2-pyridylcarbinol (α-[α-*Pyridyl*]-*benzyl alcohol*, 2-α-*hydroxybenzylpyridine*)



$C_{12}H_{11}ON$ MW, 185

Cryst. from C_6H_6–pet. ether. M.p. 82°. Very sol. EtOH, Et_2O, C_6H_6, dil. min. acids. Insol. H_2O, pet. ether. $KMnO_4$ ⟶ phenyl 2-pyridyl ketone.

B_2,H_2PtCl_6: orange-red plates from H_2O. M.p. 197° decomp. Spar. sol. cold H_2O.

Tschitschibabin, *Ber.*, 1904, **37**, 1371.

Phenyl-4-pyridylcarbinol (α-[γ-*Pyridyl*]-*benzyl alcohol*, 4-α-*hydroxybenzylpyridine*).

Cryst. from C_6H_6 or AcOEt. M.p. 126°. Very sol. EtOH, min. acids. Spar. sol. Et_2O, C_6H_6. Insol. H_2O, pet. ether. $KMnO_4$ ⟶ phenyl 4-pyridyl ketone.

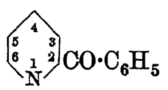
B_2,H_2PtCl_6: orange-red scales from H_2O. M.p. 205°. Spar. sol. cold H_2O.

Tschitschibabin, *Ber.*, 1904, **37**, 1371.

Phenylpyridylethane.

See Phenylethylpyridine.

Phenyl 2-pyridyl Ketone (2-*Benzoylpyridine*)



$C_{12}H_9ON$ MW, 183

Oil. B.p. 317°, 170–2°/10 mm. D_0^{20} 1·1558. Red. ⟶ phenyl-2-pyridylcarbinol.

B,HCl: cryst. from Me_2CO. M.p. 126–8°.

B_2,H_2PtCl_6: cryst. from dil. HCl. M.p. 193° decomp.

Oxime: two forms. (i) *syn.*-form. Pale yellow prisms. M.p. 150–2°. (ii) *anti*-form. Cryst. M.p. 165–7°.

Phenylhydrazone: yellow cryst. from EtOH. M.p. 136–7°. Spar. sol. cold EtOH.

Picrate: cryst. from EtOH or Me_2CO. M.p. 130°. Sol. MeOH, AcOEt. Spar. sol. C_6H_6.

Crook, McElvain, *J. Am. Chem. Soc.*, 1930, **52**, 4006.
Tschugajew, *Ber.*, 1906, **39**, 3387.
Tschitschibabin, *Chem. Zentr.*, 1902, I, 206.
Huntress, Walter, *J. Am. Chem. Soc.*, 1948, **70**, 3703.

Phenyl 3-pyridyl Ketone (*3-Benzoylpyridine*).

Cryst. M.p. 42°. B.p. 318–19°. Ox. ⟶ nicotinic acid. Red. ⟶ 3-benzylpyridine.

B,HCl: needles. M.p. 160–2°.

B_2,H_2PtCl_6: cryst. from dil. HCl. M.p. 245° decomp.

Oxime: two forms. (i) M.p. 141–3°. Sol. hot EtOH. Spar. sol. H_2O. (ii) Prisms from C_6H_6. M.p. 162–3°. Sol. EtOH. Spar. sol. Et_2O. Insol. H_2O. Sol. in dil. HCl, pptd. with Na_2CO_3 ⟶ (i).

Phenylhydrazone: m.p. 143·5°.

Picrate: clusters of needles from EtOH. M.p. 161°.

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2486.
Tschitschibabin, *Ber.*, 1903, **36**, 2711.

Phenyl 4-pyridyl Ketone (*4-Benzoylpyridine*).

Plates from H_2O, needles from pet. ether. M.p. 71·5–72·5°. B.p. 313·5–314°/742 mm. Very sol. EtOH, Et_2O. Spar. sol. hot H_2O. Red. ⟶ phenyl-4-pyridylcarbinol.

B,HCl: cryst. from EtOH–Et_2O. M.p. 195–7°.

Oxime: two forms. (i) Monoclinic prisms from EtOH. M.p. 176–7° decomp. (ii) Cryst. from EtOH. M.p. 152–5°.

Phenylhydrazone: yellow cryst. from EtOH. M.p. 181–2°.

Picrate: monoclinic cryst. from EtOH. M.p. 159–60°.

Crook, McElvain, *J. Am. Chem. Soc.*, 1930, **52**, 4006.
Tschitschibabin, *Chem. Zentr.*, 1902, I, 206.

Phenylpyridylmethane.

See Benzylpyridine.

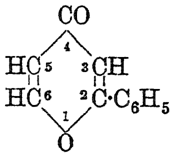
Phenylpyromellitic Acid.

See Diphenyl-2 : 3 : 5 : 6-tetracarboxylic Acid.

6-Phenyl-α-pyrone.

See 6-Phenylcoumalin.

2-Phenyl-γ-pyrone

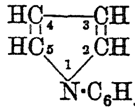


$C_{11}H_8O_2$ MW, 172

Needles from H_2O. M.p. 104°.

Borsche, Peter, *Ann.*, 1927, **453**, 158.

1-Phenylpyrrole



$C_{10}H_9N$ MW, 143

Scales with camphor-like odour. M.p. 58°. B.p. 234°, 140°/38 mm. Very sol. pet. ether. Mod. sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Insol. H_2O. Readily volatile in steam. Sublimes. Heat of comb. C_v 1283·1 Cal. Turns red in air. Unaffected by acids and alkalis. EtOH–HCl ⟶ "pine-chip" violet. Dist. through glowing tube ⟶ 2-phenylpyrrole.

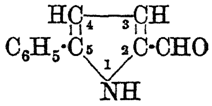
Köttnitz, *J. prakt. Chem.*, 1872, **6**, 148.
Blicke, Powers, *J. Am. Chem. Soc.*, 1944, **66**, 304.

2-Phenylpyrrole.

Plates from EtOH. M.p. 129°. B.p. 271–2°/726 mm. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Sublimes. Readily volatile in steam.

Pictet, Crépieux, *Ber.*, 1895, **28**, 1905.

5-Phenylpyrrole-2-aldehyde



$C_{11}H_9ON$ MW, 171

Needles from C_6H_6–pet. ether. M.p. 138°.

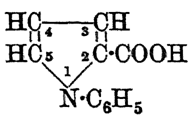
Semicarbazone: greenish plates from EtOH.Aq. M.p. 190°.

p-*Nitrophenylhydrazone*: dark red needles from EtOH. M.p. 222°.

Azine: yellow cryst. from C_6H_6. M.p. 240°.

Plancher, Rossi, Ghigi, *Gazz. chim. ital.*, 1929, **59**, 347.

1-Phenylpyrrole-2-carboxylic Acid



$C_{11}H_9O_2N$ MW, 187

Plates or needles from EtOH or C_6H_6. M.p. 166° decomp. to 1-phenylpyrrole. Very sol. EtOH, $CHCl_3$, AcOH, C_6H_6. Sol. Et_2O. Insol. cold H_2O, pet. ether. Loses CO_2 in boiling water.

Me ester: $C_{12}H_{11}O_2N$. MW, 201. Plates from EtOH.Aq. M.p. 88°. B.p. 282°. Insol. H_2O.

Et ester: $C_{13}H_{13}O_2N$. MW, 215. B.p. 289°.

Anilide: needles from EtOH.Aq. M.p. 136°.

Pictet, Steinmann, *Ber.*, 1902, **35**, 2530.

2-Phenylpyrrole-3-carboxylic Acid.

Cubes. M.p. 192–3° decomp.

Et ester: prisms. M.p. 19°. B.p. 194–5°/6 mm.

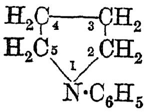
Kondo, Suzuki, *Chem. Zentr.*, 1927, II, 1029.

5-Phenylpyrrole-3-carboxylic Acid.

Cryst. from EtOH. M.p. 206–8°.

Blicke, Warzynski, Faust, Gearien, *J. Am. Chem. Soc.*, 1944, **66**, 1675.

1-Phenylpyrrolidine



$C_{10}H_{13}N$ MW, 147

Oil. B.p. 110–16°/9 mm. D^{25} 1·0260. n_D^{25} 1·5803.

B_2,H_2PtCl_6: yellow needles from EtOH–HCl. M.p. 174–5° decomp.

$B,2(COOH)_2,\frac{1}{2}H_2O$: cryst. from Me_2CO–pet. ether. M.p. 156°.

Picrate: m.p. 115–16°.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 715.

Craig, Hixon, *J. Am. Chem. Soc.*, 1930, **52**, 807.

2-Phenylpyrrolidine.

Oil. B.p. 236–8°, 115°/15 mm. n_D^{25} 1·5390. Spar. sol. H_2O giving alk. sol. Absorbs CO_2 from the air.

B,HCl: m.p. 163–4°.

$B,HAuCl_4$: yellow prisms. M.p. 110°.

B_2,H_2PtCl_6: prisms. M.p. 187–8°.

Picrate: plates. M.p. 150–1°.

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2477.

Gabriel, Colman, *Ber.*, 1908, **41**, 520.

3-Phenylpyrrolidine.

Oil. B.p. 120–2°/12 mm. Spar. sol. H_2O. Slowly absorbs CO_2 from the air.

$B,HAuCl_4$: bright yellow cryst. M.p. 137–8°.

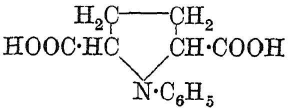
B_2,H_2PtCl_6: orange-yellow cryst. M.p. 210°.

Picrate: cryst. from EtOH. M.p. 166°.

Späth, Breusch, *Monatsh.*, 1928, **50**, 352.

Bergel, Hindley, Morrison, Rinderknecht, *J. Chem. Soc.*, 1944, 269.

1-Phenylpyrrolidine-2 : 5-dicarboxylic Acid



$C_{12}H_{13}O_4N$ MW, 235

Needles from Me_2CO–pet. ether. Decomp. at 252°. Very sol. cold EtOH, hot Me_2CO. Spar. sol. cold Et_2O, H_2O. Insol. $CHCl_3$, pet. ether, C_6H_6. Decolourises acid or alk. $KMnO_4$.

Ba salt, $8H_2O$: needles from EtOH.Aq. Very sol. H_2O. Insol. abs. EtOH.

Di-Me ester: $C_{14}H_{17}O_4N$. MW, 263. Needles from pet. ether. M.p. 88°. B.p. 225–30°/32 mm. Very sol. hot EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Insol. cold H_2O, cold EtOH, pet. ether.

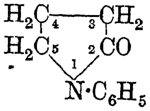
Di-Et ester: $C_{16}H_{21}O_4N$. MW, 291. Yellow oil. B.p. 227–8°/30 mm. Immiscible with H_2O.

Monoanilide: needles from $CHCl_3$–C_6H_6. Decomp. at 184°. Very sol. EtOH, Me_2CO. Spar. sol. cold H_2O, pet. ether, C_6H_6.

Le Sueur, Haas, *J. Chem. Soc.*, 1910, **97**, 177.

Le Sueur, *J. Chem. Soc.*, 1909, **95**, 276.

1-Phenylpyrrolidone-2 (N-*Phenyl-γ-butyrolactam*)



$C_{10}H_{11}ON$ MW, 161

Needles from EtOH.Aq. or C_6H_6–pet. ether, plates from H_2O. M.p. 68–9°. B.p. 180–2°/11 mm.

Lipp, Caspers, *Ber.*, 1925, **58**, 1013.

4-Phenylpyrrolidone-2 (2-*Phenyl-γ-butyrolactam*).

Hexagonal prisms from C_6H_6–pet. ether. M.p. 75–6°.

N-*Acetyl*: hexagonal plates from EtOH. M.p. 63°.

N-*Benzoyl*: prisms from C_6H_6–pet. ether. M.p. 145°.

Jackson, Kenner, *J. Chem. Soc.*, 1928, 1659.

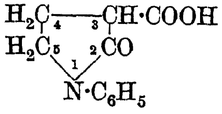
Koelsch, Stratton, *J. Am. Chem. Soc.*, 1944, **66**, 1883.

5-Phenylpyrrolidone-2 (3-*Phenyl-γ-butyrolactam*).

Needles. M.p. 91°.

Köhl, *Ber.*, 1903, **36**, 174.

1-Phenyl-2-pyrrolidone-3-carboxylic Acid

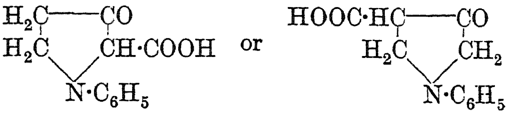


$C_{11}H_{11}O_3N$ MW, 205

M.p. 64–5°.

Küster, Grassner, *Z. physiol. Chem.*, 1925, **145**, 45.

1-Phenyl-3-pyrrolidone-2-carboxylic Acid (*1-Phenyl-3-pyrrolidone-4-carboxylic acid*)



$C_{11}H_{11}O_3N$ MW, 205

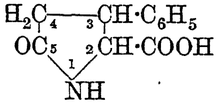
Cryst. from H_2O. M.p. 143–4°. Loses CO_2 at 170–80°. $FeCl_3$ ⟶ no col.

Et ester: $C_{13}H_{15}O_3N$. MW, 233. Needles from EtOH, plates from EtOH.Aq. M.p. 69–70°. $FeCl_3$ ⟶ dark violet col.

Phenylhydrazone: m.p. 160–1°.

de Mouilpied, *J. Chem. Soc.*, 1905, **87**, 442.

3-Phenyl-5-pyrrolidone-2-carboxylic Acid



$C_{11}H_{11}O_3N$ MW, 205

Needles and prisms from EtOH. M.p. 196·5–197·5°.

Harington, *J. Biol. Chem.*, 1925, **64**, 29.

1-Phenyl-5-pyrrolidone-3-carboxylic Acid.

Needles from H_2O, plates from EtOH.Aq. M.p. 190° slight decomp. Insol. dil. HCl.

Ag salt: needles from H_2O.

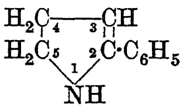
Ba salt: very sol. H_2O.

Chloride: $C_{11}H_{10}O_2NCl$. MW, 223·5. Cryst. Easily decomp. by H_2O.

Anilide: plates from EtOH.Aq. M.p. 185°. Very sol. Et_2O. Spar. sol. H_2O. Sublimes.

Anschütz, Reuter, *Ann.*, 1889, **254**, 141.

2-Phenyl-2-pyrroline



$C_{10}H_{11}N$ MW, 145

Cryst. with strong ammoniacal odour and bitter taste. M.p. 44–5°. B.p. 249°/752 mm. Spar. sol. hot H_2O. Aq. sol. reacts alkaline. Red. ⟶ 2-phenylpyrrolidine.

B,HCl: plates from Me_2CO or EtOH–Et_2O. M.p. 210° (206–7°).

B_2,H_2PtCl_6: orange plates or needles from dil. HCl. M.p. 220° decomp. (darkens at 200°).

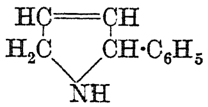
Picrate: cryst. from MeOH. M.p. 198°.

Gabriel, Colman, *Ber.*, 1908, **41**, 517.

Sonn, Podschus, Schützler, Stephani, *Ber.*, 1935, **68**, 150.

Craig, Bulbrook, Hixon, *J. Am. Chem. Soc.*, 1931, **53**, 1833.

2-Phenyl-3-pyrroline



$C_{10}H_{11}N$ MW, 145

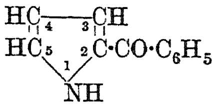
M.p. 45°. B.p. 118°/11 mm.

B,HCl: cryst. from EtOH.Aq. M.p. 240°.

Lipp, Seeles, *Ber.*, 1929, **62**, 2458.

Wohl, *Ber.*, 1901, **34**, 1922.

Phenyl 2-pyrryl Ketone (*2-Benzoylpyrrole*)



$C_{11}H_9ON$ MW, 117

Needles, plates or prisms from hot H_2O, EtOH.Aq., or pet. ether. M.p. 79°. B.p. 305°. Very sol. EtOH, AcOH, C_6H_6. Sol. pet. ether. Insol. cold H_2O. Stable to boiling KOH.

Oxime: needles from EtOH.Aq. M.p. 147°.

Oddo, Dainotti, *Gazz. chim. ital.*, 1912, **42**, i, 730.

Pictet, *Ber.*, 1904, **37**, 2797.

Phenylpyruvic Acid (*α-Ketohydrocinnamic acid*)

$C_6H_5·CH_2·CO·COOH$

$C_9H_8O_3$ MW, 164

Plates from $CHCl_3$. M.p. 157° (153–4°) decomp. Very sol. EtOH, Et_2O. Sol. hot $CHCl_3$, C_6H_6. Spar. sol. boiling H_2O. Insol. cold ligroin. Oxidises in air. Freshly prepared sol. in K_2CO_3 decolourises $KMnO_4$. Reduces boiling Fehling's and Nessler's. Salts react with semicarbazide but free acid does not. Et_2O sol. ⟶ green ring with $FeCl_3$ sol. Ac_2O ⟶ 1-acetoxycinnamic acid. Red. ⟶ benzylglycollic acid.

Na salt, H_2O: insol. EtOH. Stable in air. H_2O of cryst. remains at 100°.

Me ester: $C_{10}H_{10}O_3$. MW, 178. Needles. M.p. 75°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Oxidises in air ⟶ $C_6H_5·CHO$. *Semicarbazone*: m.p. 196°.

Et ester: $C_{11}H_{12}O_3$. MW, 192. Needles. M.p. 45°. B.p. 154·5°/15 mm., 117–21°/2·8 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Oxidises in air ⟶ $C_6H_5·CHO$. $FeCl_3$ ⟶ violet col. Ac_2O ⟶ ethyl 1-acetoxycinnamic ester. Can be separated into 3 forms, 2 enolic *cis–trans* isomerides and one ketonic:—

α. Needles from ligroin. M.p. 52°. β. B.p. 152°/15 mm. γ. M.p. 79°. α and β with $FeCl_3$ ⟶ green col. γ Gives no col. α and β give same phenylurethane of ethyl 1-hydroxycinnamic ester. All give same acetate and benzoate of 1-OH-cinnamic ester, and same ketonic derivatives. *Oxime*: $C_{11}H_{13}O_3N$. MW, 207. Prisms or needles from ligroin. M.p.

57–8°. Sol. EtOH, Et_2O, C_6H_6. Mod. sol. ligroin. Spar. sol. H_2O. *Semicarbazone*: plates from EtOH.Aq. M.p. 159–60° (167°). *Phenylhydrazone*: m.p. 89°. p-*Tolylhydrazone*: orange prisms. M.p. 72°. Sol. EtOH, Me_2CO, C_6H_6. p-*Nitrophenylhydrazone*: yellow cryst. from AcOH. M.p. 181°.

Amide: $C_9H_9O_2N$. MW, 163. M.p. 190°. Sol. hot EtOH. Insol. H_2O, Et_2O, C_6H_6. *Oxime*: needles from H_2O. M.p. 147°.

Anilide: m.p. 126°.

Oxime: $C_9H_9O_3N$. MW, 179. Needles from EtOH.Aq., or H_2O. M.p. 159° (153–67°) decomp. Sol. EtOH, Et_2O, C_6H_6.

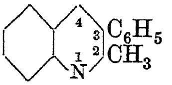
Semicarbazone: m.p. 180° decomp.

p-*Nitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 187–8° decomp.

p-*Tolylhydrazone*: yellow felted needles from EtOH, prisms from Me_2CO. M.p. 158° decomp.

Erlenmeyer, Arbenz, *Ann.*, 1904, **333**, 228.
Bougault, *Compt. rend.*, 1916, **162**, 761.
Gault, Weick, *Bull. soc. chim.*, 1922, **31**, 867.
Feist, Rauterberg, *Ber.*, 1922, **55**, 3702.
Walker, *J. Chem. Soc.*, 1925, 1862.
Kon, Watson, *J. Chem. Soc.*, 1932, 7.
Herbst, Shemin, *Organic Syntheses*, Collective Vol. II, 519.

3-Phenylquinaldine (2-*Methyl*-3-*phenylquinoline*)



$C_{16}H_{13}N$ MW, 219

Oil. B.p. 207–9°.

Picrate: yellow cryst. M.p. 170°.

Methiodide: light brown cryst. M.p. 196°.

Borsche, Vorbach, *Ann.*, 1938, **537**, 22.

4-Phenylquinaldine (2-*Methyl*-4-*phenylquinoline*).

Plates from EtOH. M.p. 100°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin, pet. ether. Sol. dil. acids with blue fluor.

B,HCl: needles from EtOH. M.p. 219°.

B_2,H_2SO_4: yellow needles from H_2O. M.p. 235°.

$ZnCl_2$ *double salt*: needles. M.p. 197°.

$CdCl_2$ *double salt*: needles. M.p. 208–10°.

$HgCl_2$ *double salt*: needles from dil. HCl. M.p. 202°.

Chloroplatinate: m.p. 235°.

Picrate: yellow needles from EtOH. M.p. 205–6°. Insol. Et_2O.

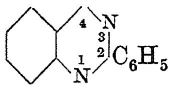
Methiodide: m.p. 205°.

Spallino, Salimei, *Gazz. chim. ital.*, 1912, **42**, i, 608.
Fischer, *J. prakt. Chem.*, 1918, **98**, 226.
Beyer, *J. prakt. Chem.*, 1886, **33**, 421.
Vasserman, *Chem. Abstracts*, 1940, **34**, 1020.

Phenylquinaldinic Acid.

See Phenylquinoline-2-carboxylic Acid.

2-Phenylquinazoline



$C_{14}H_{10}N_2$ MW, 206

Needles from EtOH. M.p. 101°. Does not boil below 300°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

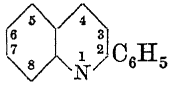
Bischler, Lang, *Ber.*, 1895, **28**, 288.

4-Phenylquinazoline.

Picrate: yellow leaflets from EtOH. M.p. 178°.

Bischler, Barad, *Ber.*, 1892, **25**, 3093.

2-Phenylquinoline



$C_{15}H_{11}N$ MW, 205

Needles from EtOH.Aq. M.p. 86° (80–1°). B.p. 363°, 310°/187 mm. Sol. Et_2O, Me_2CO, AcOEt, C_6H_6, CS_2, hot EtOH. Spar. sol. H_2O. Very spar. sol. pet. ether. Triboluminescent. $KMnO_4$ in dil. H_2SO_4 $\longrightarrow$ *N*-benzoylanthranilic acid.

Dichromate: m.p. 145–8°.

Ferrichloride: m.p. 169–70°.

B,HCl: yellow needles from alc. HCl. M.p. 163–5°.

$B_2,2HCl,AuCl_3$: needles from HCl. M.p. about 204°.

$B,HAuCl_4$: m.p. about 160°.

$B_2,H_2PtCl_6,2H_2O$: yellow needles. Decomp. about 210°.

Picrate: yellow leaflets from EtOH. M.p. 191–2°.

Methiodide: orange-red cryst. from EtOH–Et_2O. M.p. 200°.

Ethiodide: yellow prisms. M.p. 195°.

Doebner, v. Miller, *Ber.*, 1883, **16**, 1665.
Goldschmidt, *Ber.*, 1895, **28**, 986.
Doebner, Gieseke, *Ann.*, 1887, **242**, 294.
Le Fèvre, Pearson, *J. Chem. Soc.*, 1932, 2807.

3-Phenylquinoline.

Plates from Et_2O. M.p. 52°. B.p. 205–7°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. volatile in steam.

B,HCl: needles from dil. HCl. M.p. 109°.

Picrate: yellow cryst. from EtOH. M.p. 205°.

Methiodide: yellow needles from MeOH. M.p. 224°.

Ethiodide: m.p. 228°.

Friedländer, Göhring, *Ber.*, 1883, **16**, 1836.
Hübner, *Ber.*, 1908, **41**, 482.
Warren, *J. Chem. Soc.*, 1936, 1367.

4-Phenylquinoline.
Needles from Et_2O or ligroin. M.p. 61–2°. Sol. most ord. org. solvents. Insol. H_2O. Sol. dil. HCl and dil. H_2SO_4 with blue fluor.
B,HCl: needles from EtOH–Et_2O. M.p. 96–7°.
B_2,H_2SO_4: plates from H_2O. M.p. 195–6°.
$ZnCl_2$ double salt: needles from dil. HCl. M.p. 115–20°.
$CdCl_2$ double salt: needles. M.p. 120–2°.
$HgCl_2$ double salt: needles. M.p. 197–9°.
B_2,H_2PtCl_6: yellow plates. M.p. 244°.
Picrate: yellow needles from EtOH. M.p. 225°.
Methiodide: yellow needles from EtOH or H_2O. M.p. 222° decomp.

Kenner, Statham, *J. Chem. Soc.*, 1935, 301.
Koenigs, Nef, *Ber.*, 1886, **19**, 2430; 1887, **20**, 622.
Koenigs, Meimberg, *Ber.*, 1895, **28**, 1038.

5-Phenylquinoline.
Needles from EtOH.Aq. M.p. 82–3°.
Picrate: m.p. 210–11°.

Elks, Hey, *J. Chem. Soc.*, 1943, 441.

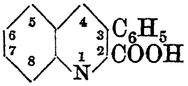
6-Phenylquinoline.
Plates from EtOH, C_6H_6 or aniline. M.p. 110–11°. B.p. 260°/77 mm. Sol. EtOH, $CHCl_3$, CS_2, C_6H_6. Spar. sol. Et_2O, pet. ether. Very spar. sol. H_2O. Spar. volatile in steam.
Picrate: m.p. 205°.
Methiodide: yellow prisms from H_2O. M.p. 194° to a red liq.

La Coste, Sorger, *Ann.*, 1885, **230**, 8.

8-Phenylquinoline.
Viscous oil with yellowish-green fluor. B.p. 283°/187 mm., 270–6°/80 mm. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Very spar. sol. H_2O.
$B_2,H_2Cr_2O_7$: reddish-yellow leaflets from H_2O. M.p. 125–6°.
Picrate: yellow needles. M.p. 200°.
Methiodide: yellow cryst. M.p. 163°. Insol. Et_2O.

La Coste, Sorger, *Ann.*, 1885, **230**, 39.
Möhlau, Berger, *Ber.*, 1893, **26**, 2004.

3-Phenylquinoline-2-carboxylic Acid (3-*Phenylquinaldinic acid*)



$C_{16}H_{11}O_2N$ MW, 249

M.p. 165° decomp. Heat. ⟶ 3-phenylquinoline.
Me ester: m.p. 82°.
Anilide: m.p. 182–3°.

Borsche, Noll, *Ann.*, 1937, **532**, 127.

4-Phenylquinoline-2-carboxylic Acid (4-*Phenylquinaldinic acid*).
Yellow needles from EtOH.Aq. M.p. 171°. At 180–90° ⟶ 4-phenylquinoline.
Chloroplatinate: yellow needles. M.p. 233–4° decomp.
Chloride: $C_{16}H_{10}ONCl$. MW, 267·5. Cryst. from ligroin. M.p. 116° ⟶ red col.

Koenigs, Jaeglé, *Ber.*, 1895, **28**, 1049.
Besthorn, *Ber.*, 1913, **46**, 2766.

2-Phenylquinoline-3-carboxylic Acid.
Needles. M.p. 226° (230° decomp.). Sol. MeOH, AcOH, $PhNO_2$. Spar. sol. C_6H_6.
Me ester: $C_{17}H_{13}O_2N$. MW, 263. M.p. 84°. B.p. 195°/13 mm.
Nitrile: needles from EtOH. M.p. 193–4°.

John, *J. prakt. Chem.*, 1931, **131**, 271.
v. Meyer, *J. prakt. Chem.*, 1914, **90**, 28.

2-Phenylquinoline-4-carboxylic Acid (2-*Phenylcinchoninic acid, cinchophene, atophan, quinophan*).
Needles from MeOH. M.p. 218°. Sol. Et_2O, hot EtOH, alkalis, warm min. acids. Insol. H_2O. The acid and several of its derivs. have wide therapeutic use as uric acid eliminants. Oxidised in human organism to 8-hydroxy-2-phenylquinoline-4-carboxylic acid. Dist. with lime ⟶ 2-phenylquinoline.
B,HCl: lemon-yellow cryst. M.p. 223°.
B,HBr: brownish-yellow cryst. M.p. 255°.
B,HI: orange cryst. M.p. 243°.
Me ester: $C_{17}H_{13}O_2N$. MW, 263. Leaflets. M.p. 61°. Bitter taste.
Et ester: $C_{18}H_{15}O_2N$. MW, 277. Cryst. from EtOH. M.p. 51°. Sol. EtOH, Et_2O. *B,HCl*: m.p. 210–11° decomp. *Picrate*: m.p. 144–5°.
Propyl ester: $C_{19}H_{17}O_2N$. MW, 291. M.p. 63–4°.
Butyl ester: $C_{20}H_{19}O_2N$. MW, 305. M.p. 56–7°.
Isobutyl ester: m.p. 39–40°.
Isoamyl ester: $C_{21}H_{21}O_2N$. MW, 319. Yellow oil. B.p. 235–40°/2 mm.
Allyl ester: atoquinol. Needles from EtOH. M.p. 30°. B.p. 265°/15 mm., 215°/0·8 mm. Sol. EtOH, Et_2O, Me_2CO. Insol. H_2O. *B,HCl*: yellow needles from EtOH. M.p. 145–7°. Insol. Et_2O.
Phenyl ester: cryst. from EtOH. M.p. 132°.
p-*Chlorophenyl ester*: m.p. 117–18°.
2-*Naphthyl ester*: yellow cryst. M.p. 130°.
Benzyl ester: yellow cryst. from EtOH. M.p. 77–8°. Sol. C_6H_6. Mod. sol. EtOH, Et_2O. Insol. H_2O.
Phenylethyl ester: m.p. 72°.
Cinnamyl ester: cryst. from Et_2O. M.p. 83°.
Salicyl ester: m.p. 188°.
Chloride: $C_{16}H_{10}ONCl$. MW, 267·5. Yellow cryst. from C_6H_6–ligroin. M.p. 81–2°. *B,HCl*: m.p. about 153° decomp.
Amide: $C_{16}H_{12}ON_2$. MW, 248. M.p. 196°.

Nitrile: $C_{16}H_{10}N_2$. MW, 230. Needles from EtOH. M.p. 140°. B.p. about 365°.

Anhydride: $C_{32}H_{20}O_3N_2$. MW, 480. Needles from C_6H_6. M.p. 185°. *Dimethiodide*: m.p. 132°.

Anilide: cryst. from EtOH. M.p. 198°.

Hydrazide: needles from EtOH. M.p. 222°. Spar. sol. Et_2O, C_6H_6.

Phenylhydrazide: cryst. from AcOH. M.p. 215°.

Azide: needles from pet. ether. M.p. 87°.

Methochloride: prisms + $2H_2O$ from H_2O. M.p. 209–10° decomp., after turning green at about 140°.

Methiodide: red cryst. from EtOH or H_2O. M.p. 160–5° decomp.

Du Puis, Lindwall, *J. Am. Chem. Soc.*, 1935, **56**, 471.
John, *Ber.*, 1926, **59**, 1447.
Rojahn, Schulten, *Arch. Pharm.*, 1926, **264**, 348.
Rosenmund, *Ber.*, 1921, **54**, 2893.
Wülfing, B.P. 325,985, (*Chem. Zentr.*, 1930, II, 93).
Boeringer Sohn, D.R.P., 485,426, (*Chem. Zentr.*, 1931, I, 853); D.R.P., 520,922, (*Chem. Zentr.*, 1931, I, 3173).
Gesellschaft für Chemische Industrie in Basel, U.S.P., 1,378,343, (*Chem. Zentr.*, 1921, IV, 514).
Bayer, D.R.P., 290,703, (*Chem. Zentr.*, 1916, I, 645).
Kalle, D.R.P., 287,304, (*Chem. Zentr.*, 1915, II, 933).

3-Phenylquinoline-4-carboxylic Acid (*3-Phenylcinchoninic acid*).

Prisms from AcOH. M.p. 277° decomp. Somewhat difficultly sol. AcOH, Me_2CO. At 290° ⟶ 3-phenylquinoline.

Me ester: needles from MeOH.Aq. M.p. 73°.

Amide: microcryst. powder from AcOH.Aq. M.p. 274°.

Anilide: needles from EtOH.Aq. M.p. 222°.

Hydrazide: platelets + $1H_2O$ from EtOH.Aq. M.p. 154°.

Hübner, *Ber.*, 1906, **39**, 983.

2-Phenylquinoline-5-carboxylic Acid.
M.p. 209–10°.

Seibert *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 2721.

2-Phenylquinoline-6-carboxylic Acid.
Cryst. from EtOH.Aq. M.p. 277°.

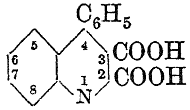
v. Braun, Brauns, *Ber.*, 1927, **60**, 1255.

2-Phenylquinoline-8-carboxylic Acid.
M.p. 158–9°.

Amide: m.p. 210–12°.

Elderfield *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 1589.

4-Phenylquinoline-2 : 3-dicarboxylic Acid (*4-Phenylacridinic acid*)



$C_{17}H_{11}O_4N$ MW, 293

M.p. 200–15°. Not obtained absolutely pure.

Claus, Nicolayson, *Ber.*, 1885, **18**, 2706.

3-Phenylquinoline-2 : 4-dicarboxylic Acid.
M.p. 271–2° decomp.

Di-Me ester: m.p. 124–5°.

Dianilide: m.p. 253–5°.

Borsche, Noll, *Ann.*, 1937, **532**, 127.

2-Phenylquinoline-3 : 4-dicarboxylic Acid.

Needles + $2H_2O$ from H_2O. M.p. 193–4°. Mod. sol. EtOH. Spar. sol. H_2O.

3-Nitrile: 2-phenyl-3-cyanocinchoninic acid. $C_{17}H_{10}O_2N_2$. MW, 274. Needles from C_6H_6. M.p. 267–8°.

Engelhard, *J. prakt. Chem.*, 1898, **57**, 471.
v. Meyer, *J. prakt. Chem.*, 1914, **90**, 23.

2-Phenylquinoline-4 : 7-dicarboxylic Acid.

Yellow cryst. powder. M.p. 200°. Sol. AcOEt. Spar. sol. EtOH, Et_2O, $CHCl_3$.

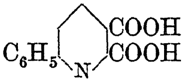
Neumann, D.R.P., 373,285, (*Chem. Zentr.*, 1923, IV, 665).

2-Phenylquinoline-4 : 8-dicarboxylic Acid.

Needles from EtOH or AcOH. Does not melt below 300°. Insol. H_2O, Et_2O, $CHCl_3$, C_6H_6, pet. ether. Heated with soda-lime ⟶ 2-phenylquinoline.

Doebner, Fettback, *Ann.*, 1894, **281**, 2.

6-Phenylquinolinic Acid (*6-Phenylpyridine-2 : 3-dicarboxylic acid*)



$C_{13}H_9O_4N$ MW, 243

M.p. 148–50° with decomp. to 6-phenylnicotinic acid.

Späth, Burger, *Monatsh.*, 1928, **49**, 270.

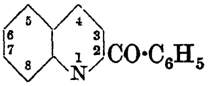
Phenyl-2-quinolylamine.
See 2-Anilinoquinoline.

Phenylquinolylethane.
See Phenylethylquinoline.

Phenylquinolylethylene
See Styrylquinoline.

***sym.*-Phenylquinolylhydrazine.**
See under 2-Quinolylhydrazine.

Phenyl 2-quinolyl Ketone (*2-Benzoylquinoline*)



$C_{16}H_{11}ON$ MW, 233

Leaflets from C_6H_6–ligroin. M.p. 111°.

Kaufmann, Dändikler, Burkhardt, *Ber.*, 1913, **46**, 2932.
Besthorn, *Ber.*, 1908, **41**, 2002.

Phenyl 4-quinolyl Ketone (4-*Benzoylquinoline.*)

Needles from H_2O. M.p. 60°. B.p. 220–4°/15 mm., 155°/0·5 mm. Sol. usual org. solvents except ligroin. Spar. sol. hot H_2O.

B,HCl: plates from EtOH. Decomp. about 204°.

Oxime: amorphous. *B,HCl*: needles from MeOH. M.p. 256° decomp.

Phenylhydrazone: yellow cryst. from C_6H_6. M.p. 239–40°.

Picrate: yellow cryst. from EtOH. M.p. 214°.

Picrolonate: yellow cryst. from EtOH. Decomp. at 174°.

Methiodide: orange leaflets. M.p. 218° (not sharp).

Kaufmann, Peyer, *Ber.*, 1913, **46**, 60.
Rabe, Pasternack, *ibid.*, 1029.

Note.—Remfrey, Decker, *Ber.*, 1908, **41**, 1008, give m.p. of phenyl 4-quinolyl ketone as 294°.

Phenyl 8-quinolyl Ketone (8-*Benzoylquinoline*).

Plates from MeOH. M.p. 94°. Very sol. EtOH, Et_2O, C_6H_6. Spar. sol. ligroin.

B_2,H_2PtCl_6: yellowish cryst. M.p. 213° decomp.

Oxime: two forms. (i) Cryst. + $1H_2O$ from EtOH. M.p. 121°. (ii) cryst. + $1H_2O$ from EtOH. M.p. 165°.

Phenylhydrazone: yellowish needles from EtOH. M.p. 190°.

Semicarbazone: cryst. from EtOH.Aq. M.p. 188°.

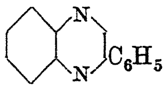
Azine: yellowish needles from C_6H_6. M.p. 287°.

Howitz, Köpke, *Ann.*, 1913, **396**, 42.

Phenylquinolylmethane.

See Benzylquinoline.

2-Phenylquinoxaline



$C_{14}H_{10}N_2$ MW, 206

Needles from EtOH. M.p. 78°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O, pet. ether. Sol. conc. H_2SO_4 and conc. HCl with yellow col.

Hinsberg, *Ann.*, 1896, **292**, 246.
Fischer, Römer, *Ber.*, 1908, **41**, 2350.

Phenyl-S Acid.

See *N*-Phenyl-1-amino-8-naphthol-4-sulphonic Acid.

Phenyl salicylate.

See Salol.

Phenylsalicylic Acid.

See 3-Hydroxydiphenyl-2-carboxylic Acid *and* 2-Hydroxydiphenyl-3-carboxylic Acid.

1-Phenylsarcosine.

See α-Methylaminophenylacetic Acid.

***N*-Phenylsarcosine** (N-*Methylphenylglycine*, N-*methylanilinoacetic acid*)

$$C_6H_5{\cdot}\overset{CH_3}{\overset{|}{N}}{\cdot}CH_2{\cdot}COOH$$

$C_9H_{11}O_2N$ MW, 165

Cryst. from Et_2O. M.p. 95–100°. Sol. EtOH. Mod. sol. H_2O.

B,HCl: prisms from conc. HCl. M.p. 210° (204°).

Me ester: $C_{10}H_{13}O_2N$. MW, 179. B.p. 140–1°/10 mm. *Methiodide*: yellowish cryst. from H_2O. M.p. 98–9°.

Et ester: $C_{11}H_{15}O_2N$. MW, 193. B.p. 156–7°/17 mm., 148°/12 mm. *Methiodide*: cryst. from H_2O. Decomp. at 129°.

Propyl ester: $C_{12}H_{17}O_2N$. MW, 207. B.p. 175°/24 mm.

Isoamyl ester: $C_{14}H_{21}O_2N$. MW, 235. B.p. 300–2°, 179–81°/17 mm.

l-*Menthyl ester*: plates from EtOH. M.p. 83·5–84·5°. $[\alpha]_D^{20}$ — 51·4° in MeOH.

Amide: $C_9H_{12}ON_2$. MW, 164. Prisms from hot H_2O. M.p. 163°. Mod. sol. EtOH.

Nitrile: $C_9H_{10}N_2$. MW, 146. M.p. 13°. B.p. 266°, 161–3°/21 mm., 148–9°/13 mm. *Methiodide*: cryst. powder from EtOH–Et_2O. M.p. 100° decomp.

Methochloride: prisms from H_2O. M.p. 194–6° decomp.

Methopicrate: needles from EtOH. M.p. 195°.

Methochloroplatinate: prisms from EtOH. M.p. 164° decomp.

Willstätter, Kahn, *Ber.*, 1904, **37**, 416.
Gault, *Bull. soc. chim.*, 1908, **3**, 373.
Knoevenagel, *Ber.*, 1904, **37**, 4083.
Öchslin, *Ann. chim.*, 1914, **1**, 243.
Warunis, Sachs, *Ber.*, 1904, **37**, 2637.

Phenylselenic Acid.

See Benzeneselenonic Acid.

Phenylselenious Acid.

See Benzeneseleninic Acid.

1-Phenylsemicarbazide

$$\underset{4}{H_2N}{\cdot}\underset{3}{CO}{\cdot}\underset{2}{NH}{\cdot}\underset{1}{NH}{\cdot}C_6H_5$$

$C_7H_9ON_3$ MW, 151

Leaflets from H_2O or EtOH.Aq. M.p. 172°. Sol. MeOH, EtOH, Me_2CO, hot H_2O. Spar. sol. Et_2O, C_6H_6, ligroin, cold H_2O. Fuming HCl ⟶ CO_2, NH_3 and phenylhydrazine.

1-*Nitroso*: yellow needles from H_2O. M.p. 126–7° decomp.

4-*Me*: m.p. 154–5°.

4-*Et*: plates from EtOH.Aq. M.p. 151°. 1-*Nitroso*: yellow needles from Me_2CO. M.p. 86·5° decomp.

1-*Acetyl*: plates from hot EtOH. M.p. 196–7° decomp.

Widman, *Ber.*, 1893, **26**, 2613 (*Footnote*).
Fischer, *Ann.*, 1877, **190**, 113.
Willstätter, Stoll, *Ber.*, 1909, **42**, 4876.
Andraca, *Chem. Abstracts*, 1942, **36**, 3802.

2-Phenylsemicarbazide

$$H_2N\cdot CO\cdot \underset{}{\overset{C_6H_5}{N}}\cdot NH_2$$

$C_7H_9ON_3$ MW, 151

Needles from EtOH. M.p. 120°. Sol. EtOH, warm H_2O. Very spar. sol. Et_2O, C_6H_6. Reduces warm Fehling's.

$B_2,Cu(NO_3)_2$: blue cryst. from H_2O. M.p. 155°.

Hydrochloride: m.p. 185–6°.

4-*Et*: $C_9H_{13}ON_3$. MW, 179. Plates from EtOH.Aq. M.p. 88°.

Picrate: yellow needles from EtOH. M.p. 163°.

Busch, Walter, *Ber.*, 1903, **36**, 1359.
Pellizzari, *Gazz. chim. ital.*, 1907, **37**, i, 621.

4-Phenylsemicarbazide

$$C_6H_5\cdot NH\cdot CO\cdot NH\cdot NH_2$$

$C_7H_9ON_3$ MW, 151

Plates from H_2O, needles from C_6H_6. M.p. 128°. Sol. EtOH, $CHCl_3$, dil. acids and alkalis. Spar. sol. hot H_2O. Insol. Et_2O.

B,HCl: prisms. M.p. 215°. Sol. H_2O, EtOH.

2-*Me*: $C_8H_{11}ON_3$. MW, 165. M.p. 93–4°.

1 : 1-*Di-Me*: $C_9H_{13}ON_3$. MW, 179. M.p. 108°.

Wheeler, *Organic Syntheses*, Collective Vol. I, 439.
Sah, Ma, *J. Chinese Chem. Soc.*, 1934, **2**, 32.

2-Phenylserine.

See 2-Hydroxy-1-amino-2-phenylpropionic Acid.

1-Phenylstearic Acid

$$CH_3\cdot[CH_2]_{15}\cdot\overset{C_6H_5}{CH}\cdot COOH$$

$C_{24}H_{40}O_2$ MW, 360

Cryst. from pet. ether. M.p. 69°. B.p. 264–5°/18 mm.

Amide: m.p. 109°.

Buu-Hoï, Cagniant, *Bull. soc. chim.*, 1943, **10**, 477.

8-Phenylstearic Acid

$$CH_3\cdot[CH_2]_8\cdot\overset{C_6H_5}{CH}\cdot[CH_2]_7\cdot COOH$$

$C_{24}H_{40}O_2$ MW, 360

M.p. 36·5–38°. B.p. 200–4°/0·09 mm. D_{25}^{25} 0·9340. n_D^{20} 1·4891.

p-*Bromophenacyl ester*: cryst. from EtOH. M.p. 83·5–84·5°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2515.

9-Phenylstearic Acid

$$CH_3\cdot[CH_2]_7\cdot\overset{C_6H_5}{CH}\cdot[CH_2]_8\cdot COOH$$

$C_{24}H_{40}O_2$ MW, 360

M.p. 40–41·5°. B.p. 199–205°/0·09 mm. D_{25}^{25} 0·9338. n_D^{20} 1·4894.

p-*Bromophenacyl ester*: cryst. from EtOH. M.p. 71–2°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2515.

α-Phenylstilbene.

See 1 : 1 : 2-Triphenylethylene.

Phenyl styryl Ketone.

See Chalkone.

Phenylstyrylmethane.

See 1 : 3-Diphenylpropylene.

Phenylsuccinic Acid

$$C_6H_5\cdot CH\cdot COOH\ \alpha$$
$$CH_2\cdot COOH\ \beta$$

$C_{10}H_{10}O_4$ MW, 194

d-.

Prisms from H_2O. M.p. 173–4°. Sol. MeOH, EtOH, Me_2CO. $[\alpha]_D^{16\cdot5}$ + 148·3° in EtOH, $[\alpha]_D^{15\cdot4}$ + 173·4° in Me_2CO.

Di-Me ester: $C_{12}H_{14}O_4$. MW, 222. B.p. 161–2°/16 mm. $[\alpha]_D^{15}$ + 169·8° in CCl_4, $[\alpha]_D^{12}$ + 140·9° in EtOH. Racemised by CH_3ONa in MeOH.

Di-Et ester: $C_{14}H_{18}O_4$. MW, 250. B.p. 166°/13 mm. $[\alpha]_D^{13}$ +103·4° in Me_2CO, $[\alpha]_D^{16}$ + 130·6° in CCl_4, $[\alpha]_D^{15\cdot5}$ + 100·8° in EtOH. Very rapidly racemised by CH_3ONa.

Anhydride: $C_{10}H_8O_3$. MW, 176. Needles from C_6H_6–pet. ether. M.p. 83·5–84·5°. Sol. $CHCl_3$, C_6H_6. Spar. sol. CCl_4, pet. ether. $[\alpha]_D^{15}$ + 100·9° in C_6H_6.

l-.

M.p. 173–4°. $[\alpha]_D^{14\cdot5}$ − 173·3° in Me_2CO, $[\alpha]_D^{12\cdot5}$ − 147·1° in MeOH. Not racemised by CH_3ONa.

Anhydride: m.p. 83·5–84·5°. $[\alpha]_D^{14}$ −100·9° in C_6H_6.

dl-.

Leaflets from H_2O. M.p. 168°. Very sol. EtOH, Et_2O, AcOH, Me_2CO. Sol. hot H_2O. Very spar. sol. $CHCl_3$, CS_2. Insol. C_6H_6, ligroin, pet. ether. $k = 1{\cdot}64 \times 10^{-4}$ at 25°.

α-*Me ester*: $C_{11}H_{12}O_4$. MW, 208. Cryst. from H_2O. M.p. 92°. Sol. MeOH, Me_2CO, $CHCl_3$, C_6H_6. $k = 1{\cdot}1 \times 10^{-4}$ at 25°. β-*Amide*: $C_{11}H_{13}O_3N$. MW, 207. Cryst. from MeOH.Aq. M.p. 119°. β-*Anilide*: needles from EtOH. M.p. 149°. β-p-*Toluidide*: m.p. 118°.

β-*Me ester*: prisms from H_2O. M.p. 102–3°. Sol. MeOH, Me_2CO, $CHCl_3$, C_6H_6. Insol. ligroin, pet. ether. $k = 0{\cdot}49 \times 10^{-4}$ at 25°. α-*Amide*: cryst. from H_2O. M.p. 145°. α-*Nitrile*: $C_{11}H_{11}O_2N$. MW, 189. Prisms from C_6H_6–pet. ether. M.p. 55°. B.p. 155–9°/10 mm. α-*Anilide*: cryst. from MeOH.Aq. M.p. 96°. α-p-*Toluidide*: m.p. 118°.

Di-Me ester: prisms from pet. ether. M.p. 57·5–58·5°. B.p. 160–2°/12 mm. Sol. MeOH, Me_2CO, $CHCl_3$, C_6H_6. Insol. H_2O.

α-*Et ester*: $C_{12}H_{14}O_4$. MW, 222. Cryst. from MeOH. M.p. 88–9°. β-*Amide*: $C_{12}H_{15}O_3N$. MW, 221. Cryst. from EtOH.Aq. M.p. 148–50°.

β-*Et ester*: cryst. from MeOH. M.p. 95–6°. α-*Amide*: cryst. from EtOH. M.p. 173°. α-*Nitrile*: $C_{12}H_{13}O_2N$. MW, 203. Oil. B.p. 176°/16 mm.

Di-Et ester: liq. B.p. 160°/10 mm.

Dichloride: $C_{10}H_8O_2Cl_2$. MW, 231. Liq. B.p. 150–1°/12 mm.

α-*Amide*: $C_{10}H_{11}O_3N$. MW, 193. Plates from H_2O. M.p. 158–9°. Sol. EtOH, Me_2CO. Spar. sol. $CHCl_3$, C_6H_6.

β-*Amide*: cryst. from H_2O. M.p. 144–5°. Very spar. sol. C_6H_6.

Diamide: $C_{10}H_{12}O_2N_2$. MW, 192. M.p. 209–10°.

α-*Nitrile*: *see* β-Cyanohydrocinnamic Acid.

Dinitrile: $C_{10}H_8N_2$. MW, 156. Cryst. from H_2O. M.p. 68–9°. Sol. EtOH, Me_2CO, $CHCl_3$.

Anhydride: needles from Et_2O. M.p. 54°. B.p. 204–6°/22 mm., 191–2°/12 mm.

Imide: $C_{10}H_9O_2N$. MW, 175. Prisms from AcOH.Aq. M.p. 90°. B.p. 205–10°/10 mm. Sol. EtOH, Me_2CO, $CHCl_3$, AcOH. Spar. sol. H_2O. N-*Phenyl*: needles from AcOEt. M.p. 138°. N-p-*Tolyl*: needles from C_6H_6. M.p. 139°.

α-*Anilide*: m.p. 175°.

β-*Anilide*: cryst. from MeOH. M.p. 170–1°.

Dianilide: m.p. 222°.

α-p-*Toluidide*: m.p. 175°.

β-p-*Toluidide*: m.p. 168–9°.

Dihydrazide: prisms from H_2O. M.p. 174·5°.

Dihydrochloride: m.p. 150°.

Wren, Williams, *J. Chem. Soc.*, 1916, **109**, 574.
Lapworth, Baker, *Organic Syntheses*, Collective Vol. I, 440.
Curtius *et al.*, *J. prakt. Chem.*, 1930, **125**, 68.
Ramart-Lucas, Papadakis, *Ann. chim.*, 1932, **18**, 32.

Phenylsulphamic Acid (*Aniline-ω-sulphonic acid*)

$C_6H_5{\cdot}NH{\cdot}SO_3H$

$C_6H_7O_3NS$ MW, 173

Plates from EtOH–Et_2O. Does not melt below 280°. Sol. H_2O. Decomp. by steam.

NH_4 *salt*: plates from EtOH.Aq. M.p. 152°. Sol. H_2O, MeOH. Spar. sol. EtOH. Insol. Et_2O, AcOEt, C_6H_6, ligroin.

Amide: $C_6H_8O_2N_2S$. MW, 172. M.p. 108·5–109°.

Dimethylamide: needles. M.p. 84–5°.

Anilide: m.p. 114°.

Traube, *Ber.*, 1890, **23**, 1654.
Paal, Kretzschmer, *Ber.*, 1894, **27**, 1244.
Bühner, *Ann.*, 1904, **333**, 288.
Battegay, Meybeck, *Compt. rend.*, 1932, **194**, 186.

Phenylsulphoneacetic Acid (*Methyl phenyl sulphone ω-carboxylic acid*)

$C_6H_5{\cdot}SO_2{\cdot}CH_2{\cdot}COOH$

$C_8H_8O_4S$ MW, 200

Needles from Et_2O. M.p. 111·5–112·5°. Sol. H_2O, EtOH, Et_2O. Mod. sol. C_6H_6. Decomp. at 160° ⟶ CO_2 + methyl phenyl sulphone.

Et ester: $C_{10}H_{12}O_4S$. MW, 228. Prisms from EtOH. M.p. 45°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

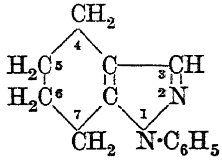
Chloride: $C_8H_7O_3ClS$. MW, 218·5. Cryst. from Et_2O. M.p. 58°.

Amide: $C_8H_9O_3NS$. MW, 199. Needles from H_2O. M.p. 156°. Sol. H_2O, EtOH. Spar. sol. Et_2O.

Nitrile: $C_8H_7O_2NS$. MW, 181. Needles. M.p. 114°. Sol. EtOH.

Troeger, Hille, *J. prakt. Chem.*, 1905, **71**, 225.
Otto, *J. prakt. Chem.*, 1884, **30**, 339.
Troeger, Vasterling, *J. prakt. Chem.*, 1905, **72**, 338.

1-Phenyltetrahydroindazole



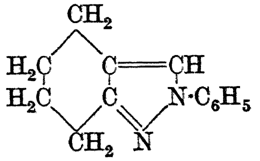
$C_{13}H_{14}N_2$ MW, 198

Plates from ligroin. M.p. 58–9°. B.p. 178°/10 mm. Sol. ord. org. solvents.

Picrate: yellow cryst. from C_6H_6. M.p. 125–6°.

Auwers, Buschmann, Heidenreich, *Ann.*, 1924, **435**, 315.

2-Phenyltetrahydroindazole



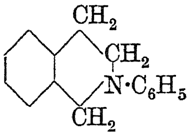
$C_{13}H_{14}N_2$ MW, 198

Prisms. M.p. 48·5–49·5°. B.p. 177°/10 mm. Sol. ord. org. solvents.

Picrate: plates from EtOH. M.p. 126·5–127·5°.

Auwers, Buschmann, Heidenreich, *Ann.*, 1924, **435**, 315.

N - Phenyl - 1 : 2 : 3 : 4 - tetrahydroiso - quinoline



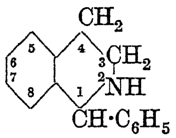
$C_{15}H_{15}N$ MW, 209

B.p. 198°/16 mm. Turns brown rapidly in air.

Picrate: yellow plates from EtOH. M.p. 120°.

v. Braun, Zobel, *Ber.*, 1923, **56**, 2152.

1 - Phenyl - 1 : 2 : 3 : 4 - tetrahydroiso - quinoline



$C_{15}H_{15}N$ MW, 209

l-.

Cryst. from pet. ether. M.p. 84°. $[\alpha]_D^{20}$ — 12·4° in $CHCl_3$.

B,HCl: cryst. from EtOH–Et_2O. M.p. 204°.

dl-.

Prisms from Et_2O. M.p. 97°. Sol. $CHCl_3$, C_6H_6. Spar. sol. EtOH, Et_2O.

N-*Me*: $C_{10}H_{13}N$. MW, 147. Needles + $1H_2O$ from EtOH.Aq. M.p. 120–30°. *Methiodide*: prisms from EtOH.Aq. M.p. 240–3°.

Bitartrate: cryst. from H_2O. M.p. 187°.

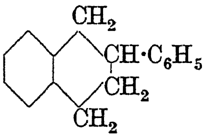
Leithe, *Monatsh.*, 1929, **53** and **54**, 961.
Freund, Bode, *Ber.*, 1909, **42**, 1761.

3 - Phenyl - 1 : 2 : 3 : 4 - tetrahydroiso - quinoline.

Cryst. M.p. 45–8°. Readily sol. ord. org. solvents.

Gabriel, *Ber.*, 1885, **18**, 3479.

2-Phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene



$C_{16}H_{16}$ MW, 208

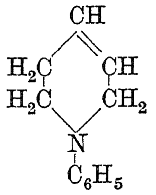
B.p. 180–1°/13 mm. D_4^{18} 1·0579. n_D^{18} 1·5980.

v. Braun, Manz, *Ann.*, 1929, **468**, 267.
Späth, *Monatsh.*, 1912, **33**, 1046.

1-Phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene-1 : 4-dicarboxylic Acid.

See Isatropic Acid.

1-Phenyl-1 : 2 : 5 : 6-tetrahydropyridine



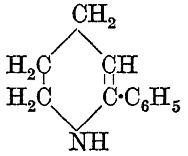
$C_{11}H_{13}N$ MW, 159

Leaflets from C_6H_6. M.p. 46°.

Picrate: yellow needles from H_2O. M.p. 146°.

Cerkownikov, Prelog, *Ber.*, 1941, **74**, 1648.

2-Phenyl-1 : 4 : 5 : 6-tetrahydropyridine



$C_{11}H_{13}N$ MW, 159

F.p. about 18°. B.p. 275–7°/751 mm.

B,HCl: cryst. from HCl. M.p. 86–7°.

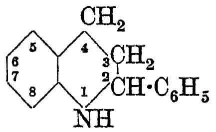
B,HAuCl₄: cryst. M.p. 118°.

B_2,H_2PtCl_6: orange-red plates. M.p. 210–11° decomp.

Picrate: yellow needles. M.p. 181°.

Gabriel, *Ber.*, 1908, **41**, 2012.
Cloke, Ayers, *J. Am. Chem. Soc.*, 1934, **56**, 2145.

2-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline



$C_{15}H_{15}N$ MW, 209

Viscous oil. B.p. 341–4°, 196–7°/12 mm. Sol. Et_2O, hot EtOH. Insol. H_2O.

N-*Me*: $C_{16}H_{17}N$. MW, 223. Needles from ligroin. M.p. 106–7°.

p-*Bromobenzenesulphonyl*: m.p. 201–2°.

Picrate: m.p. 129°.

Doebner, Miller, *Ber.*, 1886, **19**, 1198.
Freund, *Ber.*, 1904, **37**, 4669.
v. Braun, Petzold, Seemann, *Ber.*, 1922, **55**, 3785.
Oldham, Johns, *J. Am. Chem. Soc.*, 1939, **61**, 3289.

3-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Cryst. from EtOH. M.p. 83°.

B,HCl: needles from EtOH.Aq. M.p. 229°.

N-*Acetyl*: prisms from EtOH.Aq. M.p. 78°.

N-*Nitroso*: m.p. 147°.

Picrate: cryst. from EtOH. M.p. 181°.

Picrolonate: yellow powder from EtOH. M.p. 205°.

v. Braun, Petzold, Seemann, *Ber.*, 1922, **55**, 3790.

4-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Plates from EtOH.Aq. M.p. 74°.
B,HCl,1H₂O: needles. M.p. 193–4°. Sublimes at 140°.
B_2,H_2SO_4: needles. M.p. 158°. Spar. sol. H_2O.
B_2,H_2PtCl_6: m.p. 215° decomp.
N-*Me*: oil. *Picrate*: yellow needles from EtOH. M.p. 222–4°.
N-*Acetyl*: plates from EtOH. M.p. 120°.
N-*Benzoyl*: needles from Et_2O. M.p. 147°.
N-*Nitroso*: yellow needles from EtOH. M.p. 72°.
Picrate: yellow needles from EtOH. M.p. 183°.

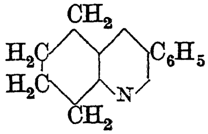
Koenigs, Meimberg, *Ber.*, 1895, **28**, 1042.
Höchst, D.R.P., 79,385.

6-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Amorphous solid. Sol. EtOH, Et_2O, hot H_2O. Insol. $CHCl_3$, C_6H_6.
$B,HCl,1\frac{1}{2}H_2O$: needles. M.p. 204°. Spar. sol. H_2O.
N-*Me*: amorphous powder from Et_2O. Sol. hot H_2O. Spar. sol. $CHCl_3$. Insol. CS_2, C_6H_6. *Methiodide*: prisms from EtOH. M.p. 194–5°. Insol. Et_2O. *Picrate*: yellow needles from EtOH. M.p. 147°.
N-*Acetyl*: needles from EtOH. M.p. 99–100°.
N-*Benzoyl*: needles from EtOH.Aq. M.p. 137°.
N-*Nitroso*: yellow cryst. from pet. ether. M.p. 111–12°.
Picrate: yellow needles from H_2O. M.p. 165°.

La Coste, *Ann.*, 1885, **230**, 20.

3-Phenyl-5 : 6 : 7 : 8-tetrahydroquinoline.



$C_{15}H_{15}N$ MW, 209

Colourless oil. B.p. 211–12°. Misc. with EtOH in all proportions.
B,HCl: m.p. 235°.
Picrolonate: yellow powder from EtOH. M.p. 201°.
Methiodide: m.p. 240–3°.

v. Braun, Petzold, Seemann, *Ber.*, 1922, **55**, 3791.

1-Phenyltetramethylene Glycol (*1-Phenyl-1 : 4-butandiol, αδ-dihydroxybutylbenzene*)

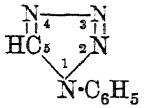
$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}C_6H_5$

$C_{10}H_{14}O_2$ MW, 166

Cryst. from Et_2O. M.p. 75°. Readily sol. org. solvents.

Marshall, Perkin, *J. Chem. Soc.*, 1891, **59**, 890.

1-Phenyl-1 : 2 : 3 : 4-tetrazole



$C_7H_6N_4$ MW, 146

Cryst. from EtOH.Aq. M.p. 65–6°. Sol. EtOH, $CHCl_3$, C_6H_6. Insol. H_2O.
$AgNO_3$ add. comp.: needles. Decomp. at 126°.
$HgCl_2$ add. comp.: needles. M.p. 147°.

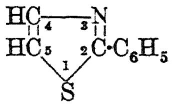
Stollé, Henke-Stark, *J. prakt. Chem.*, 1930, **124**, 281.
Freund, Paradies, *Ber.*, 1901, **34**, 3120.
Dimroth, de Montmollin, *Ber.*, 1910, **43**, 2907.

5-Phenyl-1 : 2 : 3 : 4-tetrazole.

Needles from EtOH. M.p. 215° decomp.
Mercuriacetate: needles from EtOH. M.p. 140° decomp.

Knoll, D.R.P., 521,870, (*Chem. Abstracts*, 1931, **25**, 3364).
Stollé, Henke-Stark, *J. prakt. Chem.*, 1930, **124**, 287.

2-Phenylthiazole



C_9H_7NS MW, 161

B.p. 267–9°/732 mm. (263–4°/772 mm.).
B,HCl: needles + $2H_2O$. M.p. 61–2°.
B_2,H_2PtCl_6: yellow micro-cryst. + $2H_2O$. M.p. 198–9° (173–5°) decomp.
Picrate: m.p. 124–5°.

Hubacher, *Ann.*, 1890, **259**, 234.
Bachstez, *Ber.*, 1914, **47**, 3164.

4-Phenylthiazole.

M.p. 52°. B.p. 273°. Sol. EtOH.
$B,HAuCl_4$: m.p. 174–5° decomp.
B_2,H_2PtCl_6: brownish-yellow needles + $2H_2O$. M.p. 196°.
Picrate: m.p. 164–5°.

Popp, *Ann.*, 1889, **250**, 279.

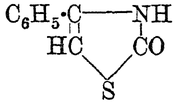
5-Phenylthiazole.

M.p. 45–6°.
$B,HAuCl_4$: m.p. 185°.
B_2,H_2PtCl_6: yellow micro-cryst. Decomp. at 281–2°.

$B_2,H_2Cr_2O_7$: needles from HCl. M.p. 108–9° decomp.
Picrate: yellow needles from EtOH. M.p. 138–9° decomp.

Bachstez, *Ber.*, 1914, **47**, 3164.

4-Phenylthiazolone

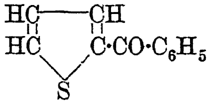


C_9H_7ONS MW, 177

M.p. 204°. Spar. sol. org. solvents. Sol. alkalis.
Hydrazone: needles from EtOH. M.p. 167–8°. N-*Acetyl*: needles from EtOH. M.p. 196–7°.

Dyckerhoff, *Ber.*, 1877, **10**, 120.
Arapides, *Ann.*, 1888, **249**, 15.
Bose, *Quart. J. Indian Chem. Soc.*, 1924, I, 60.

Phenyl 2-thienyl Ketone (*2-Benzoylthiophene*)



$C_{11}H_8OS$ MW, 188

Needles from pet. ether. M.p. 56·5–57°. B.p. 300°. Sol. EtOH, Et_2O. Insol. H_2O.
Oxime: α-*form*, needles from EtOH. M.p. 93°. *Acetyl*: m.p. 80°. β-*Form*: m.p. 113–14°. *Acetyl*: m.p. 88–9°.

Minnis, *Organic Syntheses*, 1932, XII, 62.
Steinkopf, D.R.P., 297,203, (*Chem. Zentr.*, 1917, I, 834).
Hantzsch, *Ber.*, 1891, **24**, 60.
Hartough, Kosak, *J. Am. Chem. Soc.*, 1947, **69**, 1012.

Phenylthioacetic Acid (*Thio-α-toluic acid*)

$C_6H_5{\cdot}CH_2{\cdot}COSH$ or $C_6H_5{\cdot}CH_2{\cdot}CSOH$

C_8H_8OS MW, 152

Oil. Standing in air slowly ⟶ di-phenylacetyl disulphide, m.p. 62°.
Et ester: $C_{10}H_{12}OS$. MW, 180. B.p. 170°/99 mm. D_4^{12} 1·0142.
Amide: phenylthioacetamide. C_8H_9NS. MW, 151. Cryst. from EtOH. M.p. 97·5–98°. Sol. EtOH, Et_2O. Spar. sol. hot H_2O. Dist. ⟶ benzyl cyanide.

Johnson, *J. Am. Chem. Soc.*, 1906, **28**, 1457.
Bernthsen, *Ann.*, 1877, **184**, 293.

Phenylthiocarbamic Acid (*Thiocarbanilic acid*)

$C_6H_5{\cdot}NH{\cdot}CSOH$ or $C_6H_5{\cdot}NH{\cdot}COSH$

C_7H_7ONS MW, 153

O-*Me ester*: C_8H_9ONS. MW, 167. Cryst. from EtOH. M.p. 97° (93–4°). Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, AcOH, C_6H_6. Insol. H_2O.
S-*Me ester*: leaflets from EtOH.Aq. M.p. 83–4°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Sol. conc. H_2SO_4, reppd. by H_2O.
Et ester: *see* Phenylthiourethane.
O-*Propyl ester*: $C_{10}H_{13}ONS$. MW, 195. Needles from EtOH. M.p. 48°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.
O-*Isopropyl ester*: cryst. from EtOH. M.p. 85·5°.
O-*Butyl ester*: $C_{11}H_{15}ONS$. MW, 209. Needles from Et_2O–pet. ether. M.p. 53°.
O-*Isobutyl ester*: prisms. M.p. 80·5°.
O-*Isoamyl ester*: $C_{12}H_{17}ONS$. MW, 233. Cryst. M.p. about 21°.
S-*Isoamyl ester*: needles from Et_2O. M.p. 67°.
O-*Allyl ester*: needles from ligroin. M.p. 64·5–65·5°.
O-l-*Menthyl ester*: cryst. from EtOH.Aq. M.p. 74–5°. $[\alpha]_D^{20}$ − 63·07° in EtOH.
O-*Phenyl ester*: $C_{13}H_{11}ONS$. MW, 239. Needles from $CHCl_3$. M.p. 142°. *Ag salt*: bronze-yellow cryst. from $CHCl_3$–EtOH. M.p. 186°.
S-*Phenyl ester*: needles from EtOH. M.p. 122–122·5°.
O-*Benzyl ester*: cryst. from EtOH. M.p. 82–82·5°.
S-*Benzyl ester*: prisms from C_6H_6. M.p. 96–7°.
Amide: *see* Phenylthiourea.

Bettschart, Bistrzycki, *Helv. Chim. Acta*, 1919, **2**, 131.
Schneider, Wrede, *Ber.*, 1914, **47**, 2040.
Wheeler, Barnes, *Am. Chem. J.*, 1900, **24**, 71.
Orndorff, Richmond, *Am. Chem. J.*, 1899, **22**, 456.

Phenylthioglycollic Acid (*Phenylmercaptoacetic acid, thioanisole-ω-carboxylic acid, carboxymethyl phenyl sulphide*)

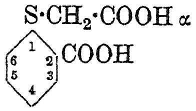
$C_6H_5{\cdot}S{\cdot}CH_2{\cdot}COOH$

$C_8H_8O_2S$ MW, 168

Needles. M.p. 61–2°. Spar. sol. cold H_2O.
Me ester: $C_9H_{10}O_2S$. MW, 182. B.p. 262–3°. D_{25}^{25} 1·1728. n_D^{25} 1·5569.
Et ester: $C_{10}H_{12}O_2S$. MW, 196. B.p. 265°, 144·5°/14 mm. D_{25}^{25} 1·1322. n_D^{25} 1·5429.
Propyl ester: $C_{11}H_{14}O_2S$. MW, 210. B.p. 270° decomp. D_{25}^{25} 1·1021. n_D^{25} 1·5348.
Butyl ester: $C_{12}H_{16}O_2S$. MW, 224. B.p. 280° decomp. D_{25}^{25} 1·0816. n_D^{25} 1·5308.
Chloride: C_8H_7OClS. MW, 186·5. B.p. 115–16°/3 mm. D_{25}^{25} 1·2581.
Amide: C_8H_9ONS. MW, 167. Needles from EtOH. M.p. 104°. Sol. EtOH. Spar. sol. H_2O, Et_2O.

Uyeda, *J. Chem. Soc. Japan*, 1931, 410.
Pummerer, *Ber.*, 1910, **43**, 1407.

Phenylthioglycollic Acid *o*-carboxylic Acid (S-*Carboxymethylthiosalicylic acid*, o-*carboxyphenylthioglycollic acid*)



$C_9H_8O_4S$ MW, 212

Needles from AcOEt. M.p. 217–18°. Sol. EtOH, Me_2CO, AcOH, AcOEt. Mod. sol. hot H_2O. Spar. sol. Et_2O. Insol. C_6H_6, ligroin.

2-*Me-ester*: $C_{10}H_{10}O_4S$. MW, 226. Cryst. from hot H_2O. M.p. 151°.

α-*Me ester*: cryst. M.p. 126–127·5°.

Di-Me ester: $C_{11}H_{12}O_4S$. MW, 240. Needles from MeOH. M.p. 52°. Insol. H_2O.

2-*Et ester*: $C_{11}H_{12}O_4S$. MW, 240. M.p. 137°.

2-*Nitrile*: *o*-cyanophenylthioglycollic acid. $C_9H_7O_2NS$. MW, 193. Yellow needles from H_2O. M.p. 140°. Sol. EtOH, Et_2O. Spar. sol. cold H_2O, C_6H_6, ligroin. *Me ester*: $C_{10}H_9O_2NS$. MW, 207. Needles from MeOH. M.p. 87–8°.

Friedländer, *Ann.*, 1907, **351**, 402.
Höchst, D.R.P., 216,725, (*Chem. Zentr.*, 1910, I, 130).
Lesser, D.R.P., 229,067, (*Chem. Zentr.*, 1911, I, 104).
Wegscheider, Joachimowitz, *Monatsh.*, 1914, **35**, 1049.
Hansch, Lindwall, *J. Org. Chem.*, 1945, **10**, 381.

Phenylthioglycollic Acid *p*-carboxylic Acid (p-*Carboxyphenylthioglycollic acid*).

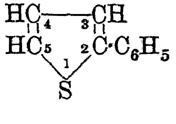
Yellow cryst. M.p. 267–9° decomp. Sol. EtOH, Me_2CO, AcOH. Spar. sol. H_2O, Et_2O, $CHCl_3$, C_6H_6, ligroin.

Di-Me ester: needles from ligroin. M.p. 63–4°.

Di-Et ester: $C_{13}H_{16}O_4S$. MW, 268. Needles from ligroin. M.p. 98°.

Friedländer, Chwala, *Monatsh.*, 1907, **28**, 279.

2-Phenylthiophene



$C_{10}H_8S$ MW, 160

Needles from EtOH. M.p. 40–1°. Sol. EtOH, AcOEt. Mod. sol. Et_2O, C_6H_6, CS_2, ligroin. Volatile in steam.

Kues, Paal, *Ber.*, 1886, **19**, 3142.

3-Phenylthiophene.

Cryst. from EtOH. M.p. 91·4–92°. B.p. 254–60°/725 mm. Sol. ord. org. solvents. Volatile in steam.

Bamberger, *Ber.*, 1897, **30**, 367.
Chrzaszczewska, *Chem. Zentr.*, 1926, II, 2905.
Voronkov, Shlyakhter, *Chem. Abstracts*, 1949, **43**, 5392.

Phenylthiosalicylic Acid.

See Diphenyl sulphide 2-carboxylic Acid.

1-Phenylthiosemicarbazide

$H_2N{\cdot}CS{\cdot}NH{\cdot}NH{\cdot}C_6H_5$
(4 3 2 1)

$C_7H_9N_3S$ MW, 167

Prisms from EtOH. M.p. 200–1° decomp. Mod. sol. hot EtOH. Spar. sol. H_2O, Et_2O, $CHCl_3$, C_6H_6.

4-N-*Me*: needles. M.p. 170° (163–4°).

4-N-*Acetyl*: prisms from EtOH. M.p. 178–9°.

4-N-*Propionyl*: prisms from EtOH. M.p. 155–6° decomp.

1-N-*Benzoyl*: cryst. from EtOH. M.p. 223°.

4-N-*Benzoyl*: prisms from EtOH. M.p. 136–7°.

1 : 4-N-*Dibenzoyl*: needles. M.p. 188°.

Fromm, Trnka, *Ann.*, 1925, **442**, 154.
Fischer, Besthorn, *Ann.*, 1882, **212**, 324.
Busch, Opfermann, Walther, *Ber.*, 1904, **37**, 2332.
Dixon, *J. Chem. Soc.*, 1889, **55**, 303; 1896, **69**, 860.

2-Phenylthiosemicarbazide

$$H_2N{\cdot}CS{\cdot}\underset{}{\overset{C_6H_5}{N}}{\cdot}NH_2$$

$C_7H_9N_3S$ MW, 167

Cryst. from hot H_2O. M.p. 153°. Sol. EtOH, hot H_2O. Spar. sol. C_6H_6.

4-N-*Me*: needles from EtOH.Aq. M.p. 91°. Sol. EtOH, Me_2CO, AcOH. Mod. sol. Et_2O, $CHCl_3$.

4-N-*Et*: prisms from EtOH. M.p. 121–2°.

Picrate: yellow cryst. from EtOH. M.p. 145°.

Pellizzari, *Gazz. chim. ital.*, 1907, **37**, 622.
Rolla, *Gazz. chim. ital.*, 1908, **38**, 345.
Dixon, *J. Chem. Soc.*, 1889, **55**, 302.

4-Phenylthiosemicarbazide

$C_6H_5{\cdot}NH{\cdot}CS{\cdot}NH{\cdot}NH_2$

$C_7H_9N_3S$ MW, 167

Plates from EtOH. M.p. 140° decomp. Spar. sol. C_6H_6. Insol. ligroin.

2-N-*Me*: plates from EtOH. M.p. 143°.

1 : 2-N-*Di-Me*: prisms from EtOH. M.p. 115°.

2-N-*Et*: needles. M.p. 109–10°.

1 : 2-N-*Dibenzoyl*: yellow cryst. from EtOH. M.p. 195°.

1 : 1 : 2 : 4-N-*Tetrabenzoyl*: yellowish-green cryst. from EtOH. M.p. 148°.

Fromm, Trnka, *Ann.*, 1925, **442**, 154.
Busch, *Ber.*, 1909, **42**, 4600.
Freund, Hempel, *Ber.*, 1895, **28**, 77.
Knorr, Köhler, *Ber.*, 1906, **39**, 3264.

Phenylthiourea

$H_2N{\cdot}CS{\cdot}NH{\cdot}C_6H_5$
3 2 1

$C_7H_8N_2S$ MW, 152

Needles from hot H_2O. M.p. 154°. Sol. EtOH.

3-N-*Me*: $C_8H_{10}N_2S$. MW, 166. Needles. M.p. 113°. Sol. EtOH.

3-N-*Di-Me*: $C_9H_{12}N_2S$. MW, 180. Prisms from EtOH. M.p. 134–5°.

3-N-*Et*: $C_9H_{12}N_2S$. MW, 180. Cryst. from EtOH or C_6H_6. M.p. 99–99·5°.

3-N-*Di-Et*: $C_{11}H_{16}N_2S$. MW, 208. Yellow oil. B.p. 182°/15 mm.

1-N-*Acetyl*: prisms from EtOH.Aq. M.p. 139° decomp.

3-N-*Acetyl*: m.p. 173° (171°). Cryst. from dil. EtOH. Mod. sol. EtOH, Et_2O. Prac. insol. H_2O. Sol. dil. alkalis.

3-N-*Propionyl*: prisms from EtOH. M.p. 129–30°.

1-N-*Benzoyl*: cryst. from EtOH. M.p. about 140°.

3-N-*Benzoyl*: needles. M.p. 148–9°.

1-N-*Phenylacetyl*: prisms from EtOH. M.p. 113–14°.

Salkowski, *Ber.*, 1891, **24**, 2728.
Gebhardt, *Ber.*, 1884, **17**, 3038.
Streiger, *Monatsh.*, 1916, **37**, 649.
Schiff, *Ann.*, 1868, **148**, 338.
Hugershoff, *Ber.*, 1899, **32**, 3659.

Phenylthiourethane

$C_6H_5{\cdot}NH{\cdot}CS{\cdot}OC_2H_5$ (i)
$C_6H_5{\cdot}NH{\cdot}CO{\cdot}SC_2H_5$ (ii)

$C_9H_{11}ONS$ MW, 181

(i) "Xanthogenanilide." Cryst. from EtOH. M.p. 71–2°. Insol. H_2O, EtOH, Et_2O. Sol. alkalis. Heat with EtI ⟶ S-ether.

(ii) Needles from EtOH. M.p. 73°. Sol. EtOH. Insol. H_2O.

Will, *Ber.*, 1882, **15**, 341.
Hofmann, *Ber.*, 1870, **3**, 774.
Jacobson, Klein, *Ber.*, 1893, **26**, 2364.
Biilmann, *Ann.*, 1906, **348**, 141.

Phenyltoluene.
See Methyldiphenyl.

Phenyltoluic Acid.
See Methyldiphenyl-carboxylic Acid.

***N*-Phenyltoluidine.**
See Methyldiphenylamine.

Phenyl-tolyl.
See Methyldiphenyl.

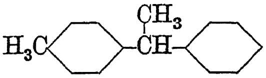
Phenyltolylamine.
See Methyldiphenylamine.

Phenyltolylcarbinol.
See Methylbenzhydrol.

Phenyl *p*-tolyl Diketone.
See 4-Methylbenzil.

***N*-Phenyltolylenediamine.**
See Amino-methyldiphenylamine.

1-Phenyl-1-*p*-tolylethane (α-*Methylphenyl*-p-*tolylmethane*)



$C_{15}H_{16}$ MW, 196

Oil. B.p. 280–2°, 154–155·8°/14 mm. $D_4^{17\cdot2}$ 0·9847. $n_D^{17\cdot2}$ 1·56590.

Auwers, *Ber.*, 1916, **49**, 2401.
Kraemer, Spilker, *Ber.*, 1891, **24**, 2788.

1-Phenyl-2-*p*-tolylethane.
See 4-Methyldibenzyl.

Phenyl tolyl Ether.
See under Cresol.

1-Phenyl-2-tolylethylene.
See Methylstilbene.

Phenyl-*o*-tolylguanidine

$C_{14}H_{15}N_3$ MW, 225

Needles from AcOH. M.p. 123–5°. Sol. EtOH, $CHCl_3$, C_6H_6. Mod. sol. H_2O.

Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 384.
Heuser, U.S.P., 1,669,242, (*Chem. Abstracts*, 1928, **22**, 2172).

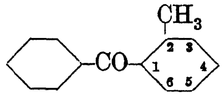
Phenyl-*p*-tolylguanidine.

Needles from ligroin. M.p. 120–2°. Sol. EtOH, C_6H_6. Mod. sol. H_2O.

Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 385.

***sym.*-Phenyltolylhydrazine.**
See Methylhydrazobenzene.

Phenyl *o*-tolyl Ketone (2-*Methylbenzophenone*)



$C_{14}H_{12}O$ MW, 196

B.p. 309·5°/762 mm., 168°/12 mm.

Oxime: phenyl-*syn*-form. M.p. 105°. Phenyl-*anti*-form: m.p. 69°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 184–90°.

Ketimide: oil. B.p. 136–7°/4 mm. $D_4^{18\cdot5}$ 1·0614. $n_D^{18\cdot5}$ 1·6065.

Smith, *Ber.*, 1891, **24**, 4047.
Goldschmidt, Stöcker, *Ber.*, 1891, **24**, 2805.

Phenyl m-tolyl Ketone (3-*Methylbenzophenone*).

Oil. B.p. 311–13°/723 mm. $D^{17.5}$ 1·088. Misc. with EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6.

Oxime: cryst. from EtOH. M.p. 100–1°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 221°.

Ador, Rilliet, *Ber.*, 1879, **12**, 2300.
Goldschmidt, Stöcker, *Ber.*, 1891, **24**, 2807.

Phenyl p-tolyl Ketone (4-*Methylbenzophenone*).

Dimorphous. Stable form, m.p. 59–60°. Metastable form, m.p. 55°. B.p. 311–12°/720 mm. Sol. Et_2O, C_6H_6. Mod. sol. EtOH.

Oxime: two forms. (i) Needles from EtOH.Aq. M.p. 153–4°. O-*Me ether*: needles from EtOH.Aq. M.p. 70·5–72°. N-*Me*: plates from ligroin. M.p. 91–2°. N-*Acetyl*: prisms. M.p. 124–5°. (ii) Needles from EtOH. M.p. 136–137·5°. O-*Me ether*: oil. B.p. 184–5°/16 mm. N-*Me*: prisms from ligroin. M.p. 113–14°.

Semicarbazone: cryst. from EtOH. M.p. 121–2°.

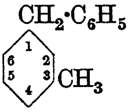
Hydrazone: cryst. from EtOH. M.p. 80–1°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 199–200°.

Ketimide: m.p. 37°. B.p. 147°/5 mm. D_4^{20} 1·0617. n_D^{20} 1·6097.

Staudinger, Goldstein, *Ber.*, 1916, **49**, 1926.
Marshall, *J. Chem. Soc.*, 1915, **107**, 516.
Meyer, *Monatsh.*, 1907, **28**, 1223.
Semper, Lichtenstadt, *Ber.*, 1918, **51**, 936.
Bruzau, *Ann. chim.*, 1934, **1**, 352.
Kuchkarov, Taukervanik, *Chem. Abstracts*, 1948, **42**, 6671.

Phenyl-m-tolylmethane (m-*Benzyltoluene*, 3-*methyldiphenylmethane*)



$C_{14}H_{14}$ MW, 182

B.p. 275°/747 mm., 268–269·5°/725 mm. $D^{17.5}$ 0·997.

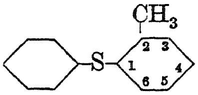
Ador, Rilliet, *Ber.*, 1879, **12**, 2300.

Phenyl-p-tolylmethane (p-*Benzyltoluene*, 4-*methyldiphenylmethane*).

B.p. 279–80°, 145·5–155·7°/13·5 mm. D^{18} 0·994. $n_\alpha^{14.7}$ 1·56922. Sol. EtOH, Et_2O, $CHCl_3$, AcOH.

Weiler, *Ber.*, 1900, **33**, 464.
Hirst, Cohen, *J. Chem. Soc.*, 1895, **67**, 828.
v. Meyer, *J. prakt. Chem.*, 1910, **82**, 539.
Rueggeberg, Cushing, Cook, *J. Am. Chem. Soc.*, 1946, **68**, 191.

Phenyl o-tolyl sulphide (2-*Methyldiphenyl sulphide*)



$C_{13}H_{12}S$ MW, 200

Oil. B.p. 304·5°/724 mm., 222·5°/100 mm. D_4^0 1·1131, D_4^{30} 1·0893.

Bourgeois, *Ber.*, 1895, **28**, 2322.
Ziegler, *Ber.*, 1890, **23**, 2471.

Phenyl m-tolyl sulphide (3-*Methyldiphenyl sulphide*).

Oil. F.p. − 6·5°. B.p. 309·5°, 164·5°/11 mm. D_4^0 1·1058, D_4^{15} 1·0937.

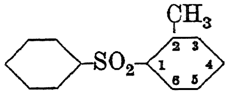
Bourgeois, *Ber.*, 1895, **28**, 2323.

Phenyl p-tolyl sulphide (4-*Methyldiphenyl sulphide*).

Oil. F.p. 15·7°. B.p. 311·5°, 167·5°/11 mm. $D^{15.7}$ 1·0900.

Bourgeois, *Ber.*, 1895, **28**, 2323.
Ziegler, *Ber.*, 1890, **23**, 2471.

Phenyl o-tolyl sulphone (2-*Methyldiphenyl sulphone*)



$C_{13}H_{12}O_2S$ MW, 232

Plates from EtOH. M.p. 80–1°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. ligroin. Insol. H_2O.

Ullmann, Lehner, *Ber.*, 1905, **38**, 734.

Phenyl m-tolyl sulphone (3-*Methyldiphenyl sulphone*).

Needles. M.p. 109°.

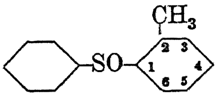
Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.

Phenyl p-tolyl sulphone (4-*Methyldiphenyl sulphone*).

Plates from EtOH. M.p. 124·5°. Spar. sol. C_6H_6, AcOH.

Michael, Adair, *Ber.*, 1878, **11**, 116.
Olivier, *Rec. trav. chim.*, 1914, **33**, 249.

Phenyl o-tolyl sulphoxide



$C_{13}H_{12}OS$ MW, 216

Prisms. M.p. 42°. B.p. 220°/11 mm.

Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.

Phenyl m-tolyl sulphoxide.

B.p. 215°/12 mm.

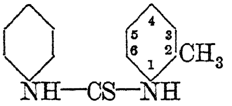
Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.

Phenyl p-tolyl sulphoxide.

M.p. 73°.

Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.

N-Phenyl-N'-o-tolylthiourea (o-*Methylthiocarbanilide*)



$C_{14}H_{14}N_2S$ MW, 242

Needles. M.p. 141–2°. Sol. EtOH, Et_2O. Spar. sol. H_2O. Phenylhydrazine ⟶ 1-phenyl-4-*o*-tolylthiosemicarbazide.

Walther, Stenz, *J. prakt. Chem.*, 1906, **74**, [2], 226.
Naunton, *J. Soc. Chem. Ind.*, 1926, **45**, 377T.

N-Phenyl-N'-m-tolylthiourea (m-*Methylthiocarbanilide*).

Prisms from EtOH. M.p. 91–2°. Very sol. Et_2O, $CHCl_3$, C_6H_6, CS_2. $AgNO_3$ in EtOH ⟶ phenyl-*m*-tolylurea.

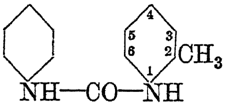
Dixon, *J. Chem. Soc.*, 1895, **67**, 557.

N-Phenyl-N'-p-tolylthiourea (p-*Methylthiocarbanilide*).

M.p. 142°. Very sol. EtOH. Sol. Et_2O. Spar. sol. H_2O. Hydrazine ⟶ 4-*p*-tolylthiosemicarbazide. Phenylhydrazine ⟶ 1-phenyl-4-*p*-tolylthiosemicarbazide.

Gebhardt, *Ber.*, 1884, **17**, 3035.

N-Phenyl-N'-o-tolylurea (o-*Methylcarbanilide*)



$C_{14}H_{14}ON_2$ MW, 226

Needles from EtOH. M.p. 212° (196°).

Sonn, *Ber.*, 1914, **47**, 2442.

N-Phenyl-N'-m-tolylurea (m-*Methylcarbanilide*).

Needles from EtOH. M.p. 173–4°.

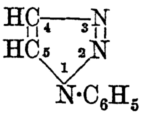
Buchka, Schachtebeck, *Ber.*, 1889, **22**, 840.

N-Phenyl-N'-p-tolylurea (p-*Methylcarbanilide*).

Needles from EtOH. M.p. 226°. Spar. sol. cold EtOH.

Mayer, *Bull. soc. chim.*, 1916, **19**, 430.

1-Phenyl-1 : 2 : 3-triazole

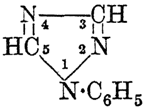


$C_8H_7N_3$ MW, 145

Plates from warm H_2O. M.p. 56°. B.p. 172–4°/18·5 mm.

Bertho, *Ber.*, 1925, **58**, 862.
Dimroth, Fester, *Ber.*, 1910, **43**, 2222.

1-Phenyl-1 : 2 : 4-triazole



$C_8H_7N_3$ MW, 145

Needles. M.p. 47°. B.p. 266°. Sol. EtOH.

B,HNO₃: needles. M.p. 141°. Spar. sol. cold H_2O.

B_2,H_2PtCl_6: yellow needles + $3H_2O$ from HCl. Also orange-red prisms + $2H_2O$. Boiling H_2O ⟶ $B_2,PtCl_4$, yellow powder. Insol. H_2O, HCl.

Picrate: cryst. from H_2O. M.p. 159°.

Pellizzari, *Gazz. chim. ital.*, 1911, **41**, ii, 20.
Widman, *Ber.*, 1893, **26**, 2615.

3-Phenyl-1 : 2 : 4-triazole.

Needles. M.p. 119·5–120°.

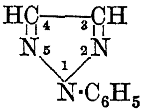
B,HCl: needles. M.p. 195°.

Acetyl: m.p. 90°.

B_2,H_2PtCl_6: yellow plates + $2H_2O$ from dil. HCl. Decomp. at 255°.

Pellizzari, *Gazz. chim. ital.*, 1911, **41**, ii, 20.
Young, Oates, *J. Chem. Soc.*, 1901, **79**, 665.

1-Phenyl-1 : 2 : 5-triazole



$C_8H_7N_3$ MW, 145

B.p. 223–4°/716 mm. Spar. sol. H_2O. Misc. with EtOH.

Jonas, Pechmann, *Ann.*, 1891, **262**, 290.
Riebsomer, *J. Org. Chem.*, 1948, **13**, 815.

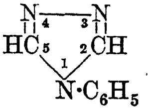
3-Phenyl-1 : 2 : 5-triazole.

Scales from EtOH.Aq. or C_6H_6. M.p. 143–5°. Exhibits acidic and basic properties.

B,HCl: cryst. powder. M.p. 140°. Insol. cold H_2O.

Oliveri-Mandalà, Coppola, *Chem. Zentr.*, 1910, II, 225.

1-Phenyl-1 : 3 : 4-triazole



$C_8H_7N_3$ MW, 145

Prisms from H_2O, needles from C_6H_6. M.p. 122°. Sol. H_2O, EtOH. Spar. sol. Et_2O. Decomp. on dist. $KMnO_4$ ⟶ 1 : 3 : 4-triazole. $CuSO_4$ ⟶ light blue ppt. $AgNO_3$ and $HgCl_2$ ⟶ white ppts.

B_2,H_2PtCl_6: needles from HCl.

Picrate: needles from EtOH. M.p. 172°.

Heller, Köhler, Gottfried, Arnold, Herrmann, *J. prakt. Chem.*, 1928, **120**, 62.
Pellizzari, Massa, *Atti accad. Lincei*, 1901, **10**, I, 366.

5-Phenyl-1 : 3 : 4-triazole.

Pale yellow plates from EtOH.Aq. M.p. 177°.

De, Roy-Choudhury, *J. Indian Chem. Soc.*, 1928, **5**, 275.

1-Phenyltricarballylic Acid

$C_6H_5·CH·COOH$
$CH·COOH$
$CH_2·COOH$

$C_{12}H_{12}O_6$ MW, 252

Two forms.

(i) Needles from Me_2CO or C_6H_6, scales from H_2O. M.p. 204° decomp. Very sol. hot H_2O, Me_2CO. Spar. sol. $CHCl_3$, boiling C_6H_6, CS_2.

(ii) Prisms + $1H_2O$ from warm H_2O. M.p. 110° (in closed capillary, m.p. 110–15°, then solidifies and remelts at 196–201°).

Tri-Et ester: b.p. 185–90°/5 mm.

Wegscheider, *Ber.*, 1911, **44**, 908.
Stobbe, Fischer, *Ann.*, 1901, **315**, 231.
Hecht, *Monatsh.*, 1903, **24**, 370.
Chatterjee, Barpujari, *J. Indian Chem. Soc.*, 1940, **17**, 292.
Weizmann, B.P. 594,182, (*Chem. Abstracts*, 1948, **42**, 2986).

1-Phenyltrimethylene Glycol (1-*Phenylpropandiol*-1 : 3, *αγ-dihydroxypropylbenzene*)

$HO·CH_2·CH_2·CH(OH)·C_6H_5$

$C_9H_{12}O_2$ MW, 152

Colourless odourless oil. B.p. 175°/11 mm. (165°/10 mm.).

Dibenzoyl: needles from ligroin. M.p. 51°.

Di-p-nitrobenzoyl: m.p. 110–110·5°.

Di-Me ether: $C_{11}H_{16}O_2$. MW, 180. B.p. 215–17° part. decomp., 94–5°/15 mm., 89–91°/12 mm. D_4^{19} 0·983.

Rupe, Müller, *Helv. Chim. Acta*, 1921, **4**, 844.
St. Pfau, Plattner, *Helv. Chim Acta*, 1932, **15**, 1265.
Straus, Berkow, *Ann.*, 1913, **401**, 155.

2-Phenyltrimethylene Glycol (2-*Phenylpropandiol*-1 : 3, *ββ′-dihydroxyisopropylbenzene*)

C_6H_5
$HO·CH_2·CH·CH_2OH$

$C_9H_{12}O_2$ MW, 152

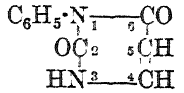
Viscous oil. B.p. 176°/13 mm. D_4^{19} 1·1161. n_D^{19} 1·54267.

Diacetyl: b.p. 162–4°/13 mm.

Methylene ether: b.p. 128–30°/13 mm. D_4^{18} 1·1111. n_D^{18} 1·53063.

Prins, *Chem. Abstracts*, 1920, **14**, 1119, 1662.

1-Phenyluracil



$C_{10}H_8O_2N_2$ MW, 188

Cryst. from H_2O. M.p. 247°. Spar. sol. H_2O, MeOH, EtOH, Py. Insol. Et_2O, AcOEt, Me_2CO, $CHCl_3$, ligroin, C_6H_6.

Buerger, Johnson, *J. Am. Chem. Soc.*, 1934, **56**, 2755.

4-Phenyluracil.

Microscopic needles and prisms from EtOH or AcOH.Aq. M.p. 269–70° decomp. Sol. 100 parts H_2O at 100°, 35 parts EtOH at 78°. Sol. AcOH, KOH. Spar. sol. Et_2O. Stable to HCl, $NH_2·NH_2$, CH_3I.

K salt: cryst. from H_2O. Decomp. above 300°.

1-*Me*: plates from EtOH. M.p. 228–30°. Sol. boiling EtOH, boiling $CHCl_3$.

1 : 3-*Di-Me*: plates from EtOH. M.p. 122–122·5°. Sol. boiling EtOH, $CHCl_3$.

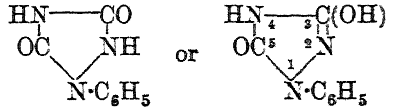
Evans, Johnson, *J. Am. Chem. Soc.*, 1930, **52**, 4999.
Johnson, Hemingway, *J. Am. Chem. Soc.*, 1915, **37**, 379.
Warmington, *J. prakt. Chem.*, 1893, **47**, 203.

5-Phenyluracil.

Microscopic plates from H_2O or EtOH. Does not melt below 350°. Sol. alkalis. Spar. sol. hot H_2O, hot EtOH.

Wheeler, Bristol, *Am. Chem. J.*, 1904, **33**, 448.

1-Phenylurazole



$C_8H_7O_2N_3$ MW, 177

Plates from H_2O. M.p. 263–4°. Sol. hot H_2O, alkalis. Spar. sol. H_2O, EtOH, Et_2O, $CHCl_3$. Behaves as monobasic acid. Reddens litmus. CO_2 liberated from carbonates. Stable to boiling alk. sols. $FeCl_3 \longrightarrow$ violet col. *O*-acyl and *O*-alkyl comps. with HCl $\longrightarrow$ *N*-comps. $PCl_5 \longrightarrow$ 1-phenyl-3 : 5-dichlorotriazole.

Ag salt: m.p. 252° decomp. Very sol. NH_4OH. Insol. dil. HNO_3.

Ba salt: plates + $2H_2O$. Decomp. at 280–300°.

2-*Me*: $C_9H_9O_2N_3$. MW, 191. Needles. M.p. 183°. Very sol. EtOH, Et_2O, alkalis. Spar. sol. H_2O. Stable to H_2SO_4 at 100°. *Ag salt*: m.p. 250° decomp. Insol. dil. HNO_3. Very sol. NH_4OH.

4-*Me*: m.p. 225°. Sol. $CHCl_3$. 2-*Acetyl*: needles from EtOH. M.p. 94–5°. 2-*Benzoyl*:

cryst. M.p. 185° Very sol. EtOH, Et_2O. Insol. H_2O. 3-*Me ether*: needles from alk. EtOH.Aq. M.p. 95°. 3-*Et ether*: cryst. from alk. EtOH.Aq. M.p. 95°.

2 : 4-*Di-Me*: $C_{10}H_{11}O_2N_3$. MW, 205. Prisms or plates from EtOH.Aq., or ligroin. M.p. 95°. Very sol. hot H_2O, EtOH, C_6H_6. Mod. sol. Et_2O. NaOH ⟶ 1-phenyl-2 : 4-dimethylsemicarbazide and 1-phenyl-2-methylurazole.

2-*Et*: $C_{10}H_{11}O_2N_3$. MW, 205. M.p. 119°. Very sol. hot H_2O, hot EtOH, Et_2O, $CHCl_3$. Spar. sol. cold H_2O, cold EtOH. *Ag salt*: m.p. 239° decomp.

2-*Me*-4-*Et*: $C_{11}H_{13}O_2N_3$. MW, 219. Cryst. from alk. EtOH.Aq. M.p. 113°.

3-*Me ether*: $C_9H_9O_2N_3$. MW, 191. Plates. M.p. 197°. Gives mono K salt. HCl ⟶ phenylurazole.

3(5)-*Et ether*: $C_{10}H_{11}O_2N_3$. MW, 205. Two forms. May be 3- and 5-Et ethers. (i) M.p. 141°. Very sol. EtOH, Et_2O, C_6H_6. Insol. H_2O, alkalis. Spar. sol. ligroin. EtOH–HCl ⟶ phenylurazole. (ii) Needles from EtOH. M.p. 152°. Sol. hot H_2O, alkalis. Behaves as monobasic acid.

3 : 5-*Di-Et ether*: $C_{12}H_{15}O_2N_3$. MW, 233. Needles from EtOH.Aq. M.p. 53°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. HCl ⟶ phenylurazole.

2-*Acetyl*: needles from H_2O, plates from C_6H_6. M.p. 175°.

2 : 4-*Diacetyl*: needles from C_6H_6. M.p. 169°. Insol. H_2O. EtOH ⟶ 2-acetyl.

2 : 4-*Dibenzoyl*: m.p. 178–80°.

Murray, Dains, *J. Am. Chem. Soc.*, 1934, **56**, 144.
Dains, Wertheim, *J. Am. Chem. Soc.*, 1920, **42**, 2308.
Brunel, Acree, *Am. Chem. J.*, 1912, **43**, 505.
Acree, *Am. Chem. J.*, 1907, **38**, 1; *Ber.*, 1903, **36**, 3139; *Ber.*, 1902, **35**, 557.

4-Phenylurazole.

Prisms from H_2O. M.p. 203°. Sol. EtOH. Spar. sol. Et_2O. Oxidising agents ⟶ deep red col.

N-*Me*: cryst. M.p. 188°. Very sol. hot H_2O, EtOH. Strongly acidic.

Thiele, Stange, *Ann.*, 1894, **283**, 45.
d'Arcangelo, *Chem. Abstracts*, 1947, **41**, 948.

Phenylurea

$$C_6H_5{\cdot}\underset{1}{NH}{\cdot}\underset{2}{CO}{\cdot}\underset{3}{NH_2}$$

$C_7H_8ON_2$ MW, 136

Needles or plates from H_2O, tablets from EtOH. M.p. 147°. Sol. hot H_2O, EtOH. Spar. sol. cold H_2O, Et_2O. Heat of comb. C_p 880 Cal. Heat ⟶ *N* : *N'*-diphenylurea. H_2SO_4 ⟶ sulphanilic and sulphocarbanilic acids. $NaNO_2$ ⟶ *N*-nitroso-*N*-phenylurea. Hydrazine ⟶ 4-phenylsemicarbazide. NaOEt ⟶ mono-Na deriv.

B,HCl: plates. M.p. 114–16° decomp. Decomp. by H_2O.

B,HNO₃: plates. M.p. 134–5° decomp. Decomp. by H_2O.

B_2,$HAuCl_4$: red needles. M.p. 147°. Decomp. by H_2O.

B_3,H_2PtCl_6: orange cryst. M.p. 173–5° decomp.

1-N-*Acetyl*: needles from hot H_2O. M.p. 167°.

2-N-*Acetyl*: needles from H_2O or EtOH. M.p. 183°.

2-N-*Benzoyl*: needles from EtOH. M.p. 210° (202°). Spar. sol. cold EtOH, Et_2O. Insol. H_2O.

1-N-*Nitroso*: $C_6H_5N(NO){\cdot}CO{\cdot}NH_2$. $C_7H_7O_2N_3$. MW, 165. Yellow needles from Et_2O–pet. ether. M.p. 95° decomp. Sol. EtOH. Spar. sol. Et_2O, C_6H_6.

Palit, *J. Indian Chem. Soc.*, 1934, **11**, 479.
Mauguin, *Ann. chim. phys.*, 1911, **22**, 346.
Pickard, Kenyon, *J. Chem. Soc.*, 1907, **91**, 902.
Schiff, *Ann.*, 1907, **352**, 83.
Stieglitz, Earle, *Am. Chem. J.*, 1903, **30**, 418.
Hoffmann, *Ann.*, 1849, **70**, 130.
Doht, Haager, *Monatsh.*, 1903, **24**, 853.
White, Bergstrom, *J. Org. Chem.*, 1942, **7**, 497.
Binder, *J. Chem. Education*, 1946, **23**, 285.

Phenylurethane (*Phenylcarbamic ethyl ester, carbanilic acid ethyl ester*)

$$C_6H_5{\cdot}NH{\cdot}CO{\cdot}OC_2H_5$$

$C_9H_{11}O_2N$ MW, 165

Needles from H_2O, plates from EtOH.Aq. M.p. 53°. B.p. 237° (slight decomp.), 152°/14 mm. Very sol. EtOH, Et_2O. Insol. cold H_2O. Heat of comb. C_p 1128·3 Cal. EtOH sol. with $AgNO_3$ + drop of NaOH ⟶ deep red to black col. NH_3 ⟶ aniline + urea. P_2O_5 ⟶ phenyl isocyanate. $R{\cdot}NH_2$ ⟶ $C_6H_5{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}R$.

K deriv.: needles. Decomp. by H_2O.

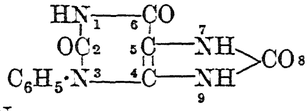
N-*Acetyl*: cryst. from ligroin. M.p. 59°. B.p. 142°/10 mm.

N-*Benzoyl*: m.p. 160–1°.

N-*Nitroso*: $C_9H_{10}O_3N_2$. MW, 194. Pale yellow needles from pet. ether. M.p. 61–2°. Sol. EtOH, Et_2O, AcOH.

Binaghi, *Gazz. chim. ital.*, 1932, **62**, 469.
Nekrassow, Melnikow, *J. prakt. Chem.*, 1930, **126**, 92.
Weizmann, Garrard, *J. Chem. Soc.*, 1920, **117**, 328.
Nijk, *Rec. trav. chim.*, 1920, **39**, 700.

3-Phenyluric Acid



$C_{11}H_8O_3N_4$ MW, 244

Cryst. + $1H_2O$ from EtOH.Aq. or AcOH.Aq. Decomp. at 270–300°. Sol. hot EtOH, AcOH. Spar. sol. cold H_2O.

1-*Me*: $C_{12}H_{10}O_3N_4$. MW, 258. Yellowish needles from AcOH. Does not melt below 340°.

1 : 7 : 9-*Tri-Me*: $C_{14}H_{14}O_3N_4$. MW, 286. Cryst. from H_2O. M.p. 229°.

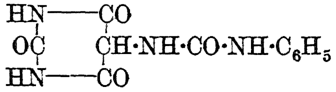
Hepner, Frenkenberg, *Helv. Chim. Acta*, 1932, **15**, 536.

9-Phenyluric Acid.

Plates + $2H_2O$ from H_2O. Sol. 120 parts H_2O at 100°. Anhyd. at 130°. Decomp. at 320°. Spar. sol. EtOH. Sol. warm H_2SO_4. Reduces $NH_3.AgNO_3$. Gives murexide reaction.

Fischer, *Ber.*, 1900, **33**, 1704.

Phenyl-ψ-uric Acid



$C_{11}H_{10}O_4N_4$ MW, 262

Aggregates of microscopic needles from H_2O. Contains H_2O of cryst. Contains $\frac{1}{2}H_2O$ at 120°, chars above this temp. Sol. 350 parts H_2O. Reduces $NH_3.AgNO_3$. Gives murexide reaction. HCl ⟶ 9-phenylurea. Na, K and NH_4 salts spar. sol. cold H_2O.

Fischer, *Ber.*, 1900, **33**, 1703.

1-Phenylvaleraldehyde (*α-Phenyl-*n*-valeraldehyde*)

$$\begin{array}{c} \quad\quad\quad\quad\quad\quad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{\cdot}CHO \end{array}$$

$C_{11}H_{14}O$ MW, 162

Oil. B.p. 122–3°/28 mm.

Semicarbazone: m.p. 115–16°.

Darzens, *Compt. rend.*, 1904, **139**, 1216.

4-Phenylvaleraldehyde

$$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CHO$$

$C_{11}H_{14}O$ MW, 162

Oil with lemon odour. B.p. 129–31°/10 mm.

p-*Nitrophenylhydrazone*: pale yellow cryst. powder from EtOH.Aq. M.p. 82–4°.

Di-Me acetal: $C_{13}H_{20}O_2$. MW, 208. B.p. 136–9°/11 mm.

v. Braun, Kruber, *Ber.*, 1912, **45**, 399.

1-Phenylvaleric Acid (*α-Phenyl-*n*-valeric acid, 1-phenylbutane-1-carboxylic acid, propylphenylacetic acid*)

$$\begin{array}{c} \quad\quad\quad\quad\quad\quad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH{\cdot}COOH \end{array}$$

$C_{11}H_{14}O_2$ MW, 178

d-.

Viscous oil. B.p. 165°/14 mm. D_4^{20} 1·047. $[\alpha]_D$ + 72·10°, $[\alpha]_D$ + 58·81° in $CHCl_3$, $[\alpha]_D$ + 33·1° in Et_2O.

l-.

$[\alpha]_D$ − 14·1° in Et_2O.

dl-.

Needles from ligroin. M.p. 58°. B.p. 280°. Resolved into active components with *l*-menthylamine.

Amide: $C_{11}H_{15}ON$. MW, 177. Cryst. from EtOH.Aq. M.p. 83–5°. Sol. org. solvents. Spar. sol. H_2O.

Nitrile: $C_{11}H_{13}N$. MW, 159. B.p. 254–5°/750 mm., 125–8°/13 mm. D^{15} 0·960. Immiscible with H_2O. Misc. with EtOH, C_6H_6. Volatile in steam.

Levene, Marker, *J. Biol. Chem.*, 1930, **88**, 53.

Bayer, D.R.P., 249,241, (*Chem. Zentr.*, 1912, II, 396).

Pickard, Yates, *J. Chem. Soc.*, 1909, **95**, 1017.

2-Phenylvaleric Acid (*β-Phenyl-*n*-valeric acid, β-ethylhydrocinnamic acid, 2-phenylbutane-1-carboxylic acid*)

$$\begin{array}{c} \quad\quad\quad C_6H_5 \\ CH_3{\cdot}CH_2{\cdot}CH{\cdot}CH_2{\cdot}COOH \end{array}$$

$C_{11}H_{14}O_2$ MW, 178

d-.

B.p. 142°/5 mm. $[\alpha]_D^{25}$ + 2·86° in C_6H_6.

l-.

B.p. 140°/4 mm. $[\alpha]_D^{25}$ − 16·5° in C_6H_6.

Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 110°/2 mm. $[\alpha]_D^{25}$ − 7·41°.

dl-.

Cryst. from Et_2O. M.p. 66°. Very sol. EtOH, Et_2O.

Me-anilide: b.p. 206°/12 mm.

Et-anilide: b.p. 214°/15 mm.

Maxim, Ioanid, *Chem. Zentr.*, 1928, II, 755.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 333; 1932, **97**, 388; 1931, **93**, 763.

3-Phenylvaleric Acid (*γ-Phenyl-*n*-valeric acid, 3-phenylbutane-1-carboxylic acid*)

$$\begin{array}{c} \quad\quad C_6H_5 \\ CH_3{\cdot}CH{\cdot}CH_2{\cdot}CH_2{\cdot}COOH \end{array}$$

$C_{11}H_{14}O_2$ MW, 178

l-.

B.p. 137°/1 mm. $[\alpha]_D^{20}$ − 1·06°.

Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 112°/1 mm. $[\alpha]_D^{25}$ − 1·23°.

dl-.

Cryst. M.p. 13°. B.p. 210°/85 mm., 170°/10 mm., 147°/1 mm. D_1^{15} 1·0554.

Chloride: $C_{11}H_{13}OCl$. MW, 196·5. B.p. 811–19°/13 mm.

Nitrile: $C_{11}H_{13}N$. MW, 159. B.p. 125–6°/13 mm.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 338.
Braun, Stuckenschmidt, *Ber.*, 1923, **56**, 1727.
Mayer, Stamm, *ibid.*, 1431.
Eijkmann, *Chem. Zentr.*, 1904, I, 1416.

4-Phenylvaleric Acid (*δ-Phenyl-*n*-valeric acid, 4-phenylbutane-*1*-carboxylic acid*)

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_{11}H_{14}O_2$ MW, 178

Plates from hot H_2O, prisms from pet. ether. M.p. 57° (61°). Sol. ord. org. solvents. Spar. sol. hot H_2O.

Me ester: $C_{12}H_{16}O_2$. MW, 192. B.p. 173°/35 mm.

Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 150°/11 mm.

Amide: $C_{11}H_{15}ON$. MW, 177. M.p. 109°.

Nitrile: $C_{11}H_{13}N$. MW, 159. B.p. 157–61°/17 mm.

Anilide: plates from MeOH. M.p. 89–90°.

Staudinger, Müller, *Ber.*, 1923, **56**, 713.
Rupe, *Ann.*, 1909, **369**, 343.
v. Braun, Deutsch, *Ber.*, 1912, **45**, 2178.

4-Phenyl-γ-valerolactone.

See under 3-Hydroxy-4-phenyl-*n*-valeric Acid.

N-Phenylvaline.

See 1-Anilinoisovaleric Acid.

3-Phenylvinylacetic Acid.

See Styrylacetic Acid.

Phenylvinylacrylic Acid.

See Styrylacrylic Acid.

1-Phenylvinyl Alcohol (*α-Hydroxystyrene*)

$CH_2{:}C(OH){\cdot}C_6H_5$

C_8H_8O MW, 120

Me ether: $C_9H_{10}O$. MW, 134. Oil with aromatic odour. B.p. 197°. D_0^{21} 1·003. n_D^{25} 1·5400. Dil. H_2SO_4 ⟶ acetophenone. Semicarbazide acetate ⟶ semicarbazone of acetophenone.

Et ether: $C_{10}H_{12}O$. MW, 148. Oil. B.p. 209–10°, 88–9°/11 mm. D_4^{20} 0·9709. $n_D^{17.5}$ 1·5304. Alc. HCl ⟶ acetophenone.

Isoamyl ether: $C_{13}H_{18}O$. MW, 190. B.p. 255–9°. D^0 0·943.

Phenyl ether: $C_{14}H_{12}O$. MW, 196. B.p. 151°/14 mm. D_{17}^{17} 1·10729.

Auwers, *Ber.*, 1911, **44**, 3520.
Moureu, *Compt. rend.*, 1903, **137**, 261.
Tiffeneau, *Compt. rend.*, 1907, **145**, 813.

2-Phenylvinyl Alcohol (*β-Hydroxystyrene*)

$C_6H_5{\cdot}CH{:}CH{\cdot}OH$

C_8H_8O MW, 120

Me ether: oil. B.p. 210–13°, 99°/13 mm. $D_4^{23.3}$ 0·9894. $n_D^{24.3}$ 1·5620. Dil. H_2SO_4 ⟶ phenylacetaldehyde.

Et ether: oil with aromatic odour. B.p. 223–6°, 105°/14 mm. $D_4^{21.4}$ 0·9714. $n_D^{21.2}$ 1·5502. Forms unstable add. comp. with Br. Boiling H_2O ⟶ phenylacetaldehyde.

Propyl ether: $C_{11}H_{14}O$. MW, 162. B.p. 238–41°. $D_0^{15.5}$ 0·966. $n_D^{15.5}$ 1·542.

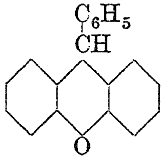
Isobutyl ether: $C_{12}H_{16}O$. MW, 176. B.p. 248–51°. D_0^{16} 0·946. n_D^{16} 1·5342.

Phenyl ether: b.p. 180°/16 mm., 157–8°/7 mm. Na + EtOH ⟶ ethylbenzene. HI ⟶ 2-phenylnaphthalene. Forms unstable bromine add. comp. Acid hyd. ⟶ phenylacetaldehyde.

Acetyl: two stereoisomeric forms. *Cis*: b.p. 68°/0 mm. D^{20} 1·073. n_D^{20} 1·5553. *Trans*: b.p. 68°/0 mm. D^{20} 1·065. n_D^{20} 1·5518. Red. ⟶ 2-phenylethyl alcohol. Catalytic red. ⟶ 2-phenylethyl acetate. Hyd. ⟶ phenylacetaldehyde.

Böeseken, Kremer, *Rec. trav. chim.*, 1931, **50**, 830.
Späth, *Monatsh.*, 1915, **36**, 6.
Auwers, Eisenlohr, *J. prakt. Chem.*, 1910, **82**, 100.
Stoermer, Biesenbach, *Ber.*, 1905, **38**, 1961.
Moureu, *Compt. rend.*, 1903, **137**, 288.
Bedoukian, *J. Am. Chem. Soc.*, 1944, **66**, 1325.

5-Phenylxanthene



$C_{19}H_{14}O$ MW, 258

Cryst. from boiling EtOH. M.p. 145°. Sol. C_6H_6. Spar. sol. EtOH, AcOH, ligroin. Insol. cold H_2SO_4.

Ruszig, *Z. angew. Chem.*, 1919, **32**, 39.
Baeyer, *Ann.*, 1907, **354**, 170.
Hubacher, *J. Am. Chem. Soc.*, 1943, **65**, 1655.

Phenyl xenyl Ketone.

See Phenylbenzophenone.

Phenyl-xylene.

See Dimethyldiphenyl.

Phenyl *p*-xylyl Ketone.

See 4-Methyldeoxybenzoin.

Phloionic Acid

$HOOC{\cdot}[CH_2]_7{\cdot}[CHOH]_2{\cdot}[CH_2]_7{\cdot}COOH$

$C_{18}H_{34}O_6$ MW, 346

Isolated from cork. Cryst. from MeOH or EtOH. M.p. 124°. Spar. sol. Et_2O, cold $CHCl_3$. Insol. cold H_2O.

Di-Me ester: $C_{20}H_{38}O_6$. MW, 374. Plates from 70% MeOH. M.p. 77–8°.

p:p′-*Dimethylcarbanilide*: needles from EtOH. M.p. 179–80°.

Zetsche *et al.*, *J. prakt. Chem.*, 1938, **150**, 140; *Helv. Chim. Acta*, 1931, **14**, 632, 846.

Phloionolic Acid

$HOOC·[CH_2]_7·[CHOH]_2·[CH_2]_7·CH_2OH$

$C_{18}H_{36}O_5$ MW, 332

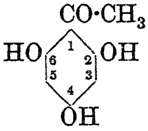
Isolated from cork. Needles from MeOH, 80% EtOH or AcOEt. M.p. 104°. An enantiomorph, with m.p. 95°, is gradually converted to stable form on standing.

Me ester: cryst. from 70% MeOH. M.p. 77°.

p:p′-*Dimethylcarbanilide*: needles from EtOH. M.p. 155–6°.

Zetsche *et al.*, *J. prakt. Chem.*, 1938, **150**, 140; *Helv. Chim. Acta*, 1931, **14**, 849.

Phloracetophenone (2 : 4 : 6-*Trihydroxyacetophenone, acetophloroglucinol*)



$C_8H_8O_4$ MW, 168

Needles from H_2O. M.p. anhyd. 219°. Sol. EtOH, Et_2O, Me_2CO, AcOH. Spar. sol. $CHCl_3$, hot C_6H_6.

2-*Me ether*: $C_9H_{10}O_4$. MW, 182. M.p. 205–7°.

4-*Me ether*: m.p. 136–7° (139–40°).

2 : 4-*Di-Me ether*: $C_{10}H_{12}O_4$. MW, 196. Constituent of fruit of *Xanthoxylum alatum*, Roxb., and volatile oil of *X. aubertia*, DC. Cryst. from EtOH. M.p. 85–8°. Sol. EtOH, Et_2O, alkalis. Insol. H_2O. *Oxime*: m.p. 108–10°. *Acetyl*: prisms from EtOH. M.p. 107°.

2 : 6-*Di-Me ether*: $C_{10}H_{12}O_4$. MW, 196. Prisms from AcOEt. M.p. 185·5°. 4-*Benzoyl*: m.p. 119°.

Tri-Me ether: $C_{11}H_{14}O_4$. MW, 210. Prisms. M.p. 103° (100°).

2-*Me-4-Et ether*: $C_{11}H_{14}O_4$. MW, 210. M.p. 56–7°.

4-*Me-2-Et ether*: m.p. 133–4°.

2 : 4-*Di-Me-6-Et ether*: $C_{12}H_{16}O_4$. MW, 224. M.p. 69–70°.

2 : 6-*Di-Me-4-Et ether*: m.p. 81–2°.

2 : 4-*Di-Et ether*: $C_{12}H_{16}O_4$. MW, 224. Needles from EtOH.Aq. M.p. 85°.

Tri-Et ether: $C_{14}H_{20}O_4$. MW, 252. Plates from EtOH.Aq. M.p. 75°.

Triacetyl: m.p. 103°.

2-*Benzoyl*: m.p. 168°.

4-*Benzoyl*: m.p. 210–11°.

Tribenzoyl: m.p. 117–18°.

Gulati, Seth, Venkataraman, *J. Chem. Soc.*, 1934, 1765; *Organic Syntheses*, 1935, XV, 70.

Tseng, *Chem. Zentr.*, 1935, II, 2826.

Sonn, *Ber.*, 1928, **61**, 2300.

Shinoda, Sato, *Chem. Abstracts*, 1928, **22**, 2947.

Howells, Little, *J. Am. Chem. Soc.*, 1932, **54**, 2452.

Kostanecki, Tambor, *Ber.*, 1899, **32**, 2262.

Phloramine.

See 5-Aminoresorcinol.

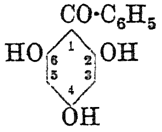
Phloraspin

$C_{23}H_{28}O_8$ MW, 432

Constituent of *Filix mas* extract. Needles from Me_2CO.Aq. M.p. 211°. Mod. sol. $CHCl_3$, Me_2CO, AcOEt, AcOH, hot EtOH, hot xylene. Spar. sol. Et_2O, C_6H_6, pet. ether, ligroin. $FeCl_3$ ⟶ reddish-brown col. in EtOH. Sol. alkalis or conc. H_2SO_4 ⟶ butyric acid. Possibly identical with flavaspidin, *q.v.*

Boehm, *Ann.*, 1903, **329**, 338.

Phlorbenzophenone (2 : 4 : 6-*Trihydroxybenzophenone*)



$C_{13}H_{10}O_4$ MW, 230

Yellow needles + $1H_2O$ from H_2O. M.p. 165°. NaOH ⟶ red col. $FeCl_3$ ⟶ brownish-red col. Reduces $NH_3.AgNO_3$ and Fehling's. Sol. EtOH, Et_2O, AcOEt. Spar. sol. $CHCl_3$, C_6H_6, ligroin.

2-*Me ether*: *see* Isocotoin.

4-*Me ether*: *see* Cotoin.

2 : 4-*Di-Me ether*: $C_{15}H_{14}O_4$. MW, 258. 6-*Acetyl*: prisms from EtOH. M.p. 83°.

2 : 6-*Di-Me ether*: plates from EtOH.Aq. M.p. 178–9°. 4-*Benzoyl*: prisms from EtOH. M.p. 172°.

4-*Me-2 : 6-Di-Et ether*: $C_{18}H_{20}O_4$. MW, 300. M.p. 82–3°.

2 : 4-*Di-Me-6-Et ether*: $C_{17}H_{18}O_4$. MW, 286. M.p. 103–4°.

4-*Benzoyl*: needles from C_6H_6. M.p. 186°.

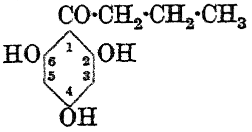
2 : 4 : 6-*Tribenzoyl*: prisms from MeOH. M.p. 125–6° (172°).

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1254.

Hoesch, *Ber.*, 1915, **48**, 1131.

Klarmann, Figdor, *J. Am. Chem. Soc.*, 1926, **48**, 804.

Phlorbutyrophenone (2 : 4 : 6-*Trihydroxybutyrophenone*)



$C_{10}H_{12}O_4$ MW, 196

Cryst. + $1H_2O$. M.p. 183° anhyd. (181°).

(?) 2-*Me ether*: $C_{11}H_{14}O_4$. MW, 210. Needles. M.p. 130°.

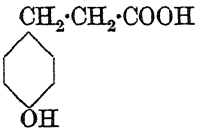
(?) 4-*Me ether*: leaflets. M.p. 113°.

2 : 4-*Di-Me ether*: $C_{12}H_{16}O_4$. MW, 224. Prisms from EtOH.Aq. M.p. 70°.

2 : 6-*Di-Me ether*: prisms from AcOEt. M.p. 107°. 4-*Benzoyl*: m.p. 86°.
4-*Benzoyl*: m.p. 164°.

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1252.

Phloretic Acid (*Phloretinic acid, p-hydrocoumaric acid, 4-hydroxyhydrocinnamic acid*)



$C_9H_{10}O_3$ MW, 166

Occurs in human urine. Prisms from Et_2O. M.p. 129–30°. Sol. EtOH, Et_2O, hot H_2O. Insol. CS_2. $k = 1{\cdot}73 \times 10^{-5}$ at 25° ($2{\cdot}03 \times 10^{-5}$). No col. with $FeCl_3$.

Me ester: $C_{10}H_{12}O_3$. MW, 180. Cryst. from Et_2O–pet. ether. M.p. 40–1°. B.p. 186–7°/17 mm.

Et ester: $C_{11}H_{14}O_3$. MW, 194. M.p. 45°. B.p. 193°/18 mm.

Amide: $C_9H_{11}O_2N$. MW, 165. Prisms. M.p. 127–8° (125°). *Acetyl deriv.*: m.p.133–4°.

Nitrile: C_9H_9ON. MW, 147. Prisms. M.p. 58–9°.

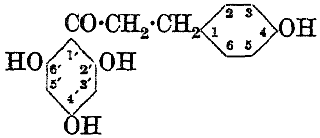
Me ether: *see* p-Methoxyhydrocinnamic Acid.

Et ether: p-ethoxyhydrocinnamic acid. $C_{11}H_{14}O_3$. MW, 194. Cryst. from H_2O. M.p. 106·5° (104°).

Carbomethoxyl: m.p. 86–7° (83–4°).

Stöhr, *Ann.*, 1884, **225**, 59.
Bayer, D.R.P., 233,551, (*Chem. Zentr.*, 1911, I, 1334).
Fischer, Nouri, *Chem. Abstracts*, 1917, **11**, 1649.
Zemplén, Csürös, Gerecs, Aczél, *Ber.*, 1928, **61**, 2492.
Bowden, Adkins, *J. Am. Chem. Soc.*, 1940, **62**, 2422.

Phloretin (*Dihydronarigenin, asebogenol, ω-p-hydroxyphenylpropiophenone*)



$C_{15}H_{14}O_5$ MW, 274

Needles from EtOH.Aq. M.p. 262–4° (274°) decomp. (varies with rate of heating). Sol. hot Me_2CO. Spar. sol. $CHCl_3$. Insol. Et_2O.

Triacetyl deriv.: m.p. 188–9°.

Tetra-acetyl: m.p. 165° (96°).

4′-*Me ether*: *see* Asebogenin.

4 : 2′ : 4′-*Tri-Me ether*: $C_{18}H_{20}O_5$. MW, 316. M.p. 109–10°. *Acetyl deriv.*: m.p. 62–3°.

4 : 2′ : 6′-*Tri-Me ether*: m.p. 142°.

β-*Glucoside*: *see* Phloridzin.

Bridel, Kramer, *Compt. rend.*, 1931, **193**, 748.
Müller, Robertson, *J. Chem. Soc.*, 1933, 1171.
Rosenmund, Rosenmund, *Ber.*, 1928, **61**, 2612.
Shinoda, Sato, Kawagoe, *Chem. Abstracts*, 1930, **24**, 604.
Fischer, Nouri, *Chem. Abstracts*, 1917, **11**, 1649.
Zemplén, Bognár, *Ber.*, 1942, **75**, 645.

Phlorhizin.
See Phloridzin.

Phlorhizoside.
See Phloridzin.

Phloridzin (*Phlorrhizin, phlorizin, phlorhizin, 2′-phloretin-β-glucoside, phlorhizoside, asebotoside, asebotin.* See formula under Phloretin above)

$C_{21}H_{24}O_{10}$ MW, 436

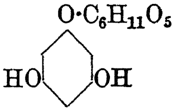
Occurs in *Micromelum teprocarpum*, Turcz. Needles. M.p. 108°. Solidifies at 138° and remelts at 170° (about 150°).

4′-*Me ether*: *see* p-Asebotin.

Tri-Me ether: $C_{24}H_{30}O_{10}$. MW, 478. M.p. 75–6°. *Tetra-acetyl deriv.*: m.p. 94–5°.

Bridel, Kramer, *Compt. rend.*, 1931, **193**, 748.
Zemplén, Csürös, Gerecs, Aczél, *Ber.*, 1928, **61**, 2486.
Müller, Robertson, *J. Chem. Soc.*, 1933, 1170.
Zemplén, Bognár, *Ber.*, 1942, **75**, 645.

Phlorin (*Phloroglucinol-β-glucoside*)

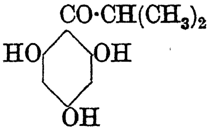


$C_{12}H_{16}O_8$ MW, 288

Cryst. M.p. 231–3°. Sol. H_2O, EtOH, MeOH. Spar. sol. Me_2CO. Insol. Et_2O, AcOEt, amyl alcohol. $[\alpha]_D^{22} - 74{\cdot}58°$.

Cremer, Seuffert, *Ber.*, 1912, **45**, 2565.

Phlorisobutyrophenone (2 : 4 : 6-*Trihydroxyisobutyrophenone*)



$C_{10}H_{12}O_4$ MW, 196

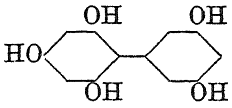
Needles + $1H_2O$ from hot H_2O. M.p. 68°, anhyd. 138–40°.

Karrer, Rosenfeld, *Helv. Chim. Acta*, 1921, **4**, 711.

Phlorizin.
See Phloridzin.

Phlorobromin.
See Octabromoacetylacetone.

Phloroglucide (*Phloroglucidol*)



Probable structure

$C_{12}H_{10}O_5$ MW, 234

Leaflets + $2H_2O$ from H_2O. Does not melt. Spar. sol. EtOH, hot H_2O. Insol. Et_2O.

Penta-acetyl: m.p. 105–7°.

Mono-Me ether: $C_{13}H_{12}O_5$. MW, 248. Cryst. from H_2O. M.p. 222–5°. *Tetra-acetyl deriv.*: m.p. 102–5°.

Penta-Me ether: $C_{17}H_{20}O_5$. MW, 304. Cryst. from EtOH. M.p. 117–20°.

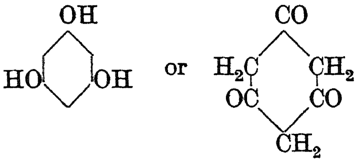
Mono-Et ether: $C_{14}H_{16}O_5$. MW, 262. Cryst. from H_2O. M.p. 165–8°.

Herzig, Kohn, *Monatsh.*, 1908, **29**, 677.
Wichelhaus, *Ber.*, 1919, **52**, 2054.
Cross, Bevan, *J. Chem. Soc.*, 1911, **99**, 1456.

Phloroglucidol.
See Phloroglucide.

Phloroglucinaldehyde.
See 2 : 4 : 6-Trihydroxybenzaldehyde.

Phloroglucinol (1 : 3 : 5-*Trihydroxybenzene*)



$C_6H_6O_3$ MW, 126

Leaflets or plates + $2H_2O$ from H_2O. M.p. 117°, 217–19° anhyd., rapid heat.; 200–9° slow heat. Sol. EtOH, Et_2O, Py. Mod. sol. H_2O. Heat of comb. C_v 617·65 Cal. $k = 4{\cdot}5 \times 10^{-10}$ at 25°. $FeCl_3$ in EtOH ⟶ bluish-violet col. Sol. in H_2O + drop NH_3.Aq. ⟶ violet-red col.

$C_6H_6O_3$,1NH_3: m.p. 88–91°.

2$C_6H_6O_3$,$(C_2H_5)_3N$: m.p. 103–4°.

Triacetyl: m.p. 104°.

Tribenzoyl: m.p. 185°.

*Mono-*p*-hydroxybenzoyl deriv.*: m.p. 218°.

Trioxime: blackens at 140°. Explodes at 155°.

Picrate: m.p. 101–3°.

Mono-Me ether: $C_7H_8O_3$. MW, 140. M.p. 78° (78–81°, 77–80°). B.p. 213°/16 mm., 188–9°/12 mm.

Di-Me ether: $C_8H_{10}O_3$. MW, 154. Cryst. from C_6H_6–ligroin. M.p. 36–8°. B.p. 172–5°/17 mm.

Tri-Me ether: $C_9H_{12}O_3$. MW, 168. Prisms from EtOH. M.p. 52·5° (54–5°). B.p. 255·5°. Sol. EtOH, Et_2O, C_6H_6.

Mono-Et ether: $C_8H_{10}O_3$. MW, 154. Leaflets + $2H_2O$ from H_2O. M.p. 84–6°. B.p. 220°/15 mm. (220–1°/30 mm.).

Di-Et ether: $C_{10}H_{14}O_3$. MW, 182. Needles from H_2O. M.p. 88–9°. B.p. 188–9°/20 mm. Volatile in steam. *Me ether*: $C_{11}H_{16}O_3$. MW, 196. B.p. 147–8°/13 mm.

Tri-Et ether: $C_{12}H_{18}O_3$. MW, 210. Cryst. M.p. 43°. B.p. 175°/24 mm. Sol. EtOH, Et_2O. Volatile in steam.

Triphenyl ether: $C_{24}H_{18}O_3$. MW, 354. Prisms from Et_2O. M.p. 112°. B.p. 290–3°/20 mm. Sol. Et_2O, C_6H_6, $CHCl_3$. Spar. sol. EtOH, AcOH, ligroin.

Dibenzyl ether: $C_{20}H_{18}O_3$. MW, 306. M.p. 62–4°. Sol. ord. org. solvents.

Tribenzyl ether: $C_{27}H_{24}O_3$. MW, 396. M.p. 39–41°. Sol. ord. org. solvents.

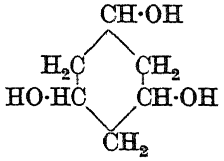
Glucoside: *see* Phlorin.

Herzig, Aigner, *Monatsh.*, 1900, **21**, 444.
Clarke, Hartman, *Organic Syntheses*, 1929, IX, 74.
Goris, Canal, *Compt. rend.*, 1935, **201**, 1435.

Phloroglucinol-dicarboxylic Acid.
See 2 : 4 : 6-Trihydroxyisophthalic Acid.

Phloroglucite.
See Phloroglucitol.

Phloroglucitol (*Phloroglucite, hexahydrophloroglucinol,* 1 : 3 : 5-*trihydroxycyclohexane, cyclohexantriol*-1 : 3 : 5)



$C_6H_{12}O_3$ MW, 132

(α) Cryst. + $2H_2O$ from H_2O. M.p. about 110° decomp. ⟶ solid remelting at 184°. Spar. sol. C_6H_6, Me_2CO.

Triacetyl: m.p. 79°.

Tri-phenylurethane: m.p. 245°.

(β) Cryst. from EtOH. M.p. 145°. Sol. H_2O. Spar. sol. C_6H_6, Me_2CO.

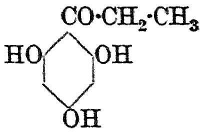
Tri-phenylurethane: m.p. 160°.

Lindemann, Baumann, *Ann.*, 1930, **477**, 78.

Phlorol.
See *o*-Ethylphenol.

Phlorone.
See *p*-Xylo-*p*-quinone.

Phlorpropiophenone (2 : 4 : 6-*Trihydroxypropiophenone*)



$C_9H_{10}O_4$ MW, 182

Needles + $1H_2O$ from H_2O. M.p. 174–5°. Sol. EtOH, warm H_2O. Alc. $FeCl_3$ ⟶ purple col.

2 : 4-*Di-Me ether* : $C_{11}H_{14}O_4$. MW, 210. Plates from EtOH. M.p. 111°. $FeCl_3$ ⟶ wine-red col.

2 : 6-*Di-Me ether* : prisms from AcOEt. M.p. 180°. No col. with $FeCl_3$. *Acetyl* : plates from EtOH.Aq. M.p. 76°. *Benzoyl* : needles from MeOH. M.p. 103°.

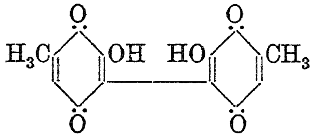
2-*Benzoyl* : prisms from MeOH. M.p. 191–2°. Alc. $FeCl_3$ ⟶ wine-red col.

4-*Benzoyl* : needles from MeOH. M.p. 193°. Alc. $FeCl_3$ ⟶ reddish-brown col.

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1245.
Howells, Little, *J. Am. Chem. Soc.*, 1932, **54**, 2452.

Phlorrhizin.
See Phloridzin.

Phœnicine (2 : 2′-*Dihydroxy*-4 : 4′-*ditoluquinone, phenicin*)



$C_{14}H_{10}O_6$ MW, 274

Pigment from *Penicillium phoeniceum* and *P. rubrum*. Yellowish brown plates from EtOH, prisms from C_6H_6. M.p. 231–2°. $FeCl_3$ ⟶ violet col. Yellowish red sols. at pH 1·8–3·5, reddish violet at pH 5·4–6·4.

Diacetyl : yellow plates from EtOH.Aq. M.p. 117–18°.

Cyclopentadiene add. comp. : $C_{24}H_{22}O_6$. Colourless needles from EtOH. M.p. 181° decomp.

Hydroquinone add. comp.: $C_{14}H_{10}O_6$, $2C_6H_6O_2$. Red plates from H_2O. M.p. 198–200°.

Tetrahydro deriv. : leucophoenicin. Colourless needles from H_2O. M.p. 247° decomp.

Posternak, *Helv. Chim. Acta*, 1938, **21**, 1326.
Friedheim, *ibid.*, 1464.
Curtin, Fitzgerald, Reilly, *Biochem. J.*, 1940, **34**, 1605.
Posternak, Ruelius, Tcherniak, *Helv. Chim. Acta*, 1943, **26**, 2031.

Phonopyrrole.
See Hæmopyrrole.

Phorone (2 : 6-*Dimethyl*-2 : 5-*heptadienone*-4, *di-isopropylideneacetone*)

$(CH_3)_2C{:}CH{\cdot}CO{\cdot}CH{:}C(CH_3)_2$

$C_9H_{14}O$ MW, 138

Yellowish-green prisms. M.p. 28°. B.p. 197·2°/743·3 mm. D_4^{20} 0·8850. n_D^{20} 1·49982.

Oxime : m.p. 48°. B.p. 218°.

Semicarbazone : m.p. 221°.

2 : 4-*Dinitrophenylhydrazone* : m.p. 118°.

Auwers, Eisenlohr, *J. prakt. Chem.*, 1911, **84**, 76.
Francis, *J. Chem. Soc.*, 1927, 2898.
I.G., D.R.P., 483,823, (*Chem. Abstracts*, 1930, **24**, 2146).
Sugden, *J. Chem. Soc.*, 1928, 412.
Pereira, *Chem. Abstracts*, 1910, **4**, 2275.
Claisen, *Ann.*, 1876, **180**, 4.

Phoronic Acid (3-*Keto*-1 : 1 : 5 : 5-*tetramethylpimelic acid*)

$(CH_3)_2C{\cdot}COOH$
CH_2
CO
CH_2
$(CH_3)_2C{\cdot}COOH$

$C_{11}H_{18}O_5$ MW, 230

Prisms from EtOH.Aq. M.p. 184° decomp. Sol. EtOH. Spar. sol. hot H_2O.

Me ester : $C_{12}H_{20}O_5$. MW, 244. Prisms from MeOH. M.p. 105–6°.

Di-Me ester : $C_{13}H_{22}O_5$. MW, 258. Prisms from pet. ether. M.p. 32°.

Di-Et ester : $C_{15}H_{26}O_5$. MW, 286. M.p. 33° (125°).

Di-lactone (*anhydride*) : $C_{11}H_{16}O_4$. MW, 212. M.p. 138° (132°).

Toivonen, *Chem. Zentr.*, 1928, II, 39.
Milikan, *Rec. trav. chim.*, 1912, **31**, 287.

Phosgene.
See Carbonyl chloride.

Phosphaniline.
See Phenylphosphine.

Phosphinobenzene.
See under Phenylphosphorous Acid.

Phosphobenzene (*Diphosphenyl*)

$C_6H_5{\cdot}P{:}P{\cdot}C_6H_5$

$C_{12}H_{10}P_2$ MW, 216

Yellow powder. M.p. 149–50°. Sol. hot C_6H_6. Insol. EtOH, Et_2O, hot H_2O.

Köhler, Michaelis, *Ber.*, 1877, **10**, 812.

Phosphoric Acid. *Organic derivatives.*

Tri-Me ester : $C_3H_9O_4P$. MW, 140. B.p. 197°, 97°/36 mm., 85°/24 mm. D^{22} 1·200.

Tri-Et ester : $C_6H_{15}O_4P$. MW, 182. B.p. 215°, 190°/445 mm., 161°/188 mm., 146°/112 mm., 123°/50 mm., 103°/25 mm., 98–98·5°/8–10 mm. D_0^{19} 1·0725. $n_D^{17 \cdot 1}$ 1·40674.

Tripropyl ester : $C_9H_{21}O_4P$. MW, 224. B.p. 138°/47 mm., 133·5°/22 mm., 120·5–121·5°/8–10 mm. D^{22} 1·007.

Tri-isopropyl ester : b.p. 218–20°/763 mm., 136°/68 mm., 95–6°/8–10 mm.

Tributyl ester : $C_{12}H_{27}O_4$. MW, 266. B.p. 160–2°/15 mm.

Tri-isobutyl ester : b.p. 192°, 119–29°/8–12 mm. D^{22} 0·965.

Tri-n-*amyl ester*: $C_{15}H_{33}O_4P$. MW, 308. B.p. 158–63°/6 mm.
Monohexadecyl ester: $C_{16}H_{35}O_4P$. MW, 322. M.p. 74°.
Triallyl ester: $C_9H_{15}O_4P$. MW, 218. B.p. 157°/44 mm.
Phenyl ester: *see* Triphenyl phosphate *and under* Phenol.
Naphthyl ester: *see under* Naphthol.
Dibenzyl ester: $C_{14}H_{15}O_4P$. MW, 278. M.p. 78–9°.
Tribenzyl ester: $C_{21}H_{21}O_4P$. MW, 368. M.p. 64°.
Dianilide: m.p. 214–16°.
Trianilide: m.p. 212–13° (208–10°).
Di-o-*toluidide*: m.p. 120°.
Di-p-*toluidide*: m.p. 195°.
Tri-o-*toluidide*: m.p. 236°.
Tri-p-*toluidide*: m.p. 192–4°.
Tri-benzylamide: m.p. 98°.
Tri-1-*naphthylamide*: m.p. 216°.
Tri-2-*naphthylamide*: m.p. 170°.

Adler, Gottlieb, U.S.P., 1,983,588, (*Chem. Abstracts*, 1935, **29**, 817).
Harlay, *Chem. Abstracts*, 1935, **29**, 2506.
Chem. Fabrik v. Heyden, B.P. 398,659, (*Chem. Abstracts*, 1934, **28**, 1362).
Noller, Dutton, *J. Am. Chem. Soc.*, 1933, **55**, 424.

Phosphorus triethyl.
See Triethylphosphine.

Phrenosin.
See Cerebron.

Phrenosinic Acid.
See Cerebronic Acid.

o-Phthalaldehyde (1 : 2-*Dialdehydobenzene*)

$C_8H_6O_2$ MW, 134

(1) Cryst. from ligroin. M.p. 53·2°.
(2) Yellow cryst. M.p. 56–7°.
Sol. H_2O, ord. org. solvents. Spar. sol. pet. ether. Volatile in steam. $CrO_3 \longrightarrow$ phthalic acid.
Di-phenylhydrazone: m.p. 190–1°.
Tetra-acetate: m.p. 126–7°.

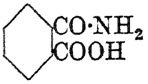
Thiele, Günther, *Ann.*, 1906, **347**, 107.
Seekles, *Rec. trav. chim.*, 1923, **42**, 706.
Bayer, D.R.P., 121,788, (*Chem. Zentr.*, 1901, II, 70).
Chaudhuri, *J. Am. Chem. Soc.*, 1942, **64**, 365.

m-Phthalaldehyde.
See Isophthalaldehyde.

p-Phthalaldehyde.
See Terephthalaldehyde.

o-Phthalaldehydic Acid.
See o-Aldehydobenzoic Acid.

Phthalamic Acid (*Phthalic acid monoamide*, o-*carbamylbenzoic acid*)

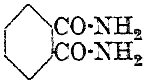


$C_8H_7O_3N$ MW, 165

Prisms. M.p. 148–9°. Mod. sol. EtOH, hot H_2O. Spar. sol. Et_2O, C_6H_6. Insol. ligroin. Heat of comb. C_p 850·7 Cal. $k = 1{\cdot}6 \times 10^{-4}$ at 25°. Heat at 155° $\longrightarrow$ phthalimide.
Nitrile: *see under* o-Cyanobenzoic Acid.
2 : 4-*Dinitrophenylhydrazone*: m.p. 298–9°.

Fodor, F.P., 636,846, (*Chem. Abstracts*, 1929, **23**, 608).
Chesnais, Can. P., 282,407, (*Chem. Abstracts*, 1928, **22**, 4133).
Aschan, *Ber.*, 1886, **19**, 1402.

Phthalamide (*Phthalic acid diamide*)

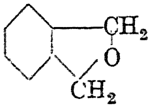


$C_8H_8O_2N_2$ MW, 164

Cryst. M.p. 219–20° slow heat. $\longrightarrow$ phthalimide + NH_3. Spar. sol. H_2O, EtOH. Heat of comb. C_p 291·7 Cal.

Hoogewerff, van Dorp, *Rec. trav. chim.*, 1892, **11**, 100.
Aschan, *Ber.*, 1886, **19**, 1399.

Phthalan (o-*Xylylene oxide*, 2 : 5-*dihydro*-3 : 4-*benzfuran*, *isocoumaran*)

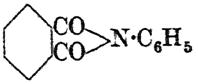


C_8H_8O MW, 120

Oil with odour of benzaldehyde. B.p. 192°. Volatile in steam. D_4^0 1·098. Alk. $KMnO_4 \longrightarrow$ phthalic acid.

Ludwig, *Ber.*, 1907, **40**, 3062.

Phthalanil (N-*Phenylphthalimide*)

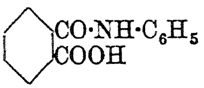


$C_{14}H_9O_2N$ MW, 223

Needles from EtOH. M.p. 210° (208°, 206°, 203°). Sol. $CHCl_3$. Insol. H_2O. Sublimes.

Cornillot, *Ann. chim.*, 1927, **8**, 177.
Jolles, *Gazz. chim. ital.*, 1935, **65**, 1224.
Warren, Briggs, *Ber.*, 1931, **64**, 29.
Das, Sarker, *J. Indian Chem. Soc.*, 1934, **11**, 709.
Poraĭ-Koshitz, *Chem. Abstracts*, 1935, **29**, 131, 6590.
See also Sherrill, Schaeffer, Shoyer, *J. Am. Chem. Soc.*, 1928, **50**, 477.

Phthalanilic Acid (*Phthalic acid monoanilide*)



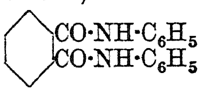
$C_{14}H_{11}O_3N$ MW, 241

Needles from EtOH. M.p. 170° ⟶ phthanil.

Anhydride: *see* Phthalanil.

Warren, Briggs, *Ber.*, 1931, **64**, 29.
Das, Sarker, *J. Indian Chem. Soc.*, 1934, **11**, 709.
Poraĭ-Koshitz, *Chem. Abstracts*, 1935, **29**, 131, 6590.
See also Sherrill, Schaeffer, Shoyer, *J. Am. Chem. Soc.*, 1928, **50**, 477.

Phthalanilide (N : N′-*Diphenylphthalamide, phthalic acid dianilide*)



$C_{20}H_{16}O_2N_2$ MW, 316

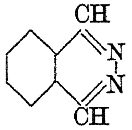
Needles from EtOH. M.p. 251° (245–50°, 231° decomp.). Spar. sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, AcOH. Heat of comb. C_p 2383·2 Cal.

Tingle, Cram, *Am. Chem. J.*, 1907, **37**, 603.
Grimmel, Morgan, *J. Am. Chem. Soc.*, 1946, **68**, 539.

Phthalanone.

See Phthalide.

Phthalazine (4 : 5-*Benzdiazine*, β-*phenodiazine*)



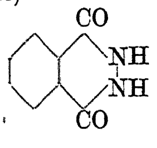
$C_8H_6N_2$ MW, 130

Needles from Et_2O. M.p. 90–1°. B.p. about 315–17° decomp., 189°/29 mm., 175°/17 mm. Sol. H_2O, EtOH, C_6H_6. Spar. sol. Et_2O. Insol. ligroin.

B,HCl: m.p. 231° decomp.
B,HI: m.p. 203°.
B,HAuCl$_4$: m.p. 200°.
Methiodide: m.p. 235–40°.
Ethiodide: m.p. 204–10°.
Benzylchloride: m.p. 97–9°.
Picrate: m.p. 208–10°.

Paul, *Ber.*, 1899, **32**, 2015.

1 : 4-Phthalazinedione (*Diketophthalazine, phthaloylhydrazine*)

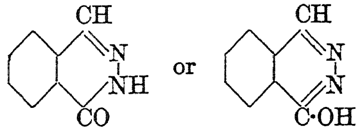


$C_8H_6O_2N_2$ MW, 162

M.p. 342–4°.

Barber, Wragg, *J. Chem. Soc.*, 1947, 1331.

Phthalazone



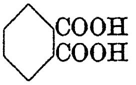
$C_8H_6ON_2$ MW, 146

Needles from H_2O. M.p. 183–4°. B.p. 337°. Sol. H_2O, EtOH, C_6H_6. Sublimes in prisms.

N-*Acetyl*: m.p. 135°.
N-*Me*: $C_9H_8ON_2$. MW, 160. M.p. 114° (111–12°).
O-*Me*: needles. M.p. 60–1°.
N-*Et*: $C_{10}H_{10}ON_2$. MW, 174. M.p. 55° (67–8°). B.p. 295°.
O-*Et*: m.p. 29–31°.

Rothenburg, *J. prakt. Chem.*, 1895, **51**, 147.
Paul, *Ber.*, 1899, **32**, 2020.

Phthalic Acid (*Benzene*-o-*dicarboxylic acid*)



$C_8H_6O_4$ MW, 166

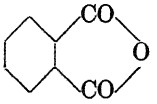
Plates from hot H_2O. M.p. 231° (rapid heat.), 191° (in sealed tube). Mod. sol. H_2O, EtOH. Spar. sol. Et_2O. Insol. $CHCl_3$. Heat of comb. C_p 771·6 Cal., C_v 779·3 Cal. k (first) = 1·3 × 10^{-3} at 25°; (second) = 3·9 × 10^{-6} at 18°.

Aniline salt: m.p. 158°.
Pyridine acid salt: m.p. 86°.
Me ester: $C_9H_8O_4$. MW, 180. M.p. 85° (82·5°). k = 6·56 × 10^{-4} at 25°.
Di-Me ester: $C_{10}H_{10}O_4$. MW, 194. B.p. 282°. $D_4^{20\cdot7}$ 1·1905. $n_D^{20\cdot7}$ 1·515. Heat of comb. C_p 1120·4 Cal., C_v 1120·1 Cal.
Me-Et ester: $C_{11}H_{12}O_4$. MW, 208. B.p. 285–7° (281–2°).
Et ester: $C_{10}H_{10}O_4$. MW, 194. M.p. 2°. D_4^{22} 1·1877. n_D^{22} 1·509. k = 5·51 × 10^{-4} at 25°.
Di-Et ester: *see* Diethyl phthalate.
Dibutyl ester: $C_{16}H_{22}O_4$. MW, 278. B.p. 212–15°. D^{21} 1·043–1·050.
d-*Isobutyl ester*: $C_{12}H_{14}O_4$. MW, 222. M.p. 46–7°. $[\alpha]_D^{20}$ + 38·4° in EtOH.
dl-*Isobutyl ester*: cryst. from pet. ether. M.p. 56–7°.
Di-isobutyl ester: b.p. 182–4°/10 mm.
Dihexadecyl ester: $C_{40}H_{70}O_4$. MW, 614. M.p. 43°.
Cyclohexyl ester: $C_{14}H_{16}O_4$. MW, 248. M.p. 99°.
Di-cyclohexyl ester: $C_{20}H_{26}O_4$. MW, 330. M.p. 66°.
Phenyl ester: $C_{14}H_{10}O_4$. MW, 242. Needles from C_6H_6–ligroin. M.p. 103°.
Diphenyl ester: $C_{20}H_{14}O_4$. MW, 318. Prisms from EtOH. M.p. 73° (70°). B.p. 250–7°/14 mm.
Di-1-naphthyl ester: $C_{28}H_{18}O_4$. MW, 418. M.p. 155°.

Benzyl ester: $C_{15}H_{12}O_4$. MW, 256. M.p. 106–7°.
Dibenzyl ester: $C_{22}H_{18}O_4$. MW, 346. M.p. 42–3°. B.p. 277°/15 mm., 274°/12 mm.
Di-p-nitrobenzyl ester: m.p. 154–5°.
Difluoride: *see* Phthaloyl fluoride.
Dichloride: *see* Phthaloyl chloride.
Monoamide: *see* Phthalamic Acid.
Diamide: *see* Phthalamide.
Mononitrile: *see* o-Cyanobenzoic Acid.
Dinitrile: *see* Phthalonitrile.
Monoanilide: *see* Phthalanilic Acid.
Dianilide: *see* Phthalanilide.
Anhydride: *see* Phthalic Anhydride.
Imide: *see* Phthalimide.
Mono-phenylhydrazide: m.p. 165–6°.
Di-phenylhydrazide: m.p. 161°.
Cyclic hydrazide: *see* 1 : 4-Phthalazinedione.

Pickard, Kenyon, *J. Chem. Soc.*, 1913, **103**, 1937; 1914, **105**, 1126.
Miller, *Chem. Zentr.*, 1914, I, 790.
Buchsweiler, D.R.P., 232,818, (*Chem. Zentr.*, 1911, I, 1090).
Reid, *J. Am. Chem. Soc.*, 1917, **39**, 1249.
Dennstedt, Hassler, D.R.P., 203,848, (*Chem. Zentr.*, 1908, II, 1750).

Phthalic Anhydride



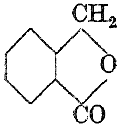
$C_8H_4O_3$ MW, 148

Needles from EtOH or C_6H_6. M.p. 131·61°. B.p. 295·09° (285·1°), 284·54°/751·4 mm., 283·75°/739 mm. Sol. EtOH. Spar. sol. Et_2O, H_2O. Sublimes. Sp. gr. 1·527. Intermediate for dyestuffs of the pyronine or phthalein class (Fluorescein, Rhodamine, Eosine, Phloxine, etc.) and for synthesis of anthraquinones, glyptal resins, and many other industrial products.
Oxime: *see* Phthaloxime.
Mono-2-pyridylhydrazide: m.p. 208°.
Mono-2-quinolylhydrazide: m.p. 236°.

Punnett, U.S.P., 1,978,506, (*Chem. Abstracts*, 1935, **29**, 176).
National Aniline, B.P. 415,748, (*Chem. Abstracts*, 1935, **29**, 819); U.S.P., 2,004,586, (*ibid.*, 5128).
Douglass, Jones, U.S.P., 2,013,727, (*Chem. Abstracts*, 1935, **29**, 6903).
Buylla, Pertierra, *Chem. Abstracts*, 1933, **27**, 1624.
Timmermans, Burrul, *Chimie et industrie*, 1931, Suppl. No., 196.
Marti, *Bull. soc. chim. Belg.*, 1930, **39**, 590.
Andrews, *Ind. Eng. Chem.*, 1921, **13**, 167.
Gibbs, *Ind. Eng. Chem.*, 1920, **12**, 1017 (*Bibl.*).

Phthalidanil.
See N-Phenylphthalimidine.

Phthalide (*1-Phthalanone, o-hydroxymethylbenzoic acid lactone, 1(2)-isobenzfuranone, 3:4-benzfuranone-2*)

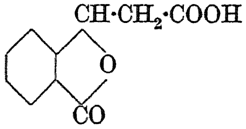


$C_8H_6O_2$ MW, 134

(i) Needles or plates from H_2O. M.p. 75° (73–4°). Stable. (ii) M.p. 65·8° (65°). Labile. Sol. EtOH, Et_2O, hot H_2O. $D_4^{99·1}$ 1·1636. $n_D^{99·1}$ 1·536. Heat of comb. C_p 885·1 Cal., C_v 884·7 Cal.

Jaeger, U.S.P., 1,889,961, (*Chem. Abstracts*, 1933, **27**, 1640).
Kalle, D.R.P., 267,596, (*Chem. Zentr.*, 1914, I, 199).
Gardner, Naylor, *Organic Syntheses*, 1936, XVI, 71.

Phthalide-3-acetic Acid

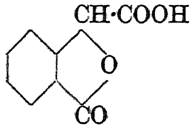


$C_{10}H_8O_4$ MW, 192

Cryst. + $1H_2O$ from H_2O. M.p. 151·5°. Sol. EtOH, hot H_2O.
Me ester: $C_{11}H_{10}O_4$. MW, 206. M.p. 62°.
Et ester: $C_{12}H_{12}O_4$. MW, 220. M.p. 76°.

Gabriel, Michael, *Ber.*, 1877, **10**, 1558.
Roth, *Ber.*, 1914, **47**, 1598.

Phthalide-3-carboxylic Acid

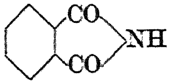


$C_9H_6O_4$ MW, 178

Leaflets from C_6H_6–AcOH. M.p. 153° (151°). Sol. H_2O, EtOH, Et_2O, AcOH.
Me ester: $C_{10}H_8O_4$. MW, 192. Needles from ligroin. M.p. 57° (53–4°).
Amide: $C_9H_7O_3N$. MW, 177. Cryst. from H_2O. M.p. 185·5°.

Fries, *Ann.*, 1904, **334**, 358.
Tscherniac, *J. Chem. Soc.*, 1916, **109**, 1240.
Ruhemann, *J. Chem. Soc.*, 1910, **97**, 2030.

Phthalimide



$C_8H_5O_2N$ MW, 147

Needles from H_2O, prisms from AcOH, leaflets by sublimation. M.p. 233–5° (238°). Sol. hot AcOH. Insol. C_6H_6, ligroin. Forms *N*-metallic derivs.

N-*Acetyl*: m.p. 133–5°.

N-*Chloro*: chlorylphthalimide. $C_8H_4O_2NCl$. MW, 181·5. M.p. 183–5°.

N-*Bromo*: $C_8H_4O_2NBr$. MW, 226. Yellow cryst. from C_6H_6. M.p. 206–7°.

N-*Me*: *see N*-Methylphthalimide.

N-*Et*: $C_{10}H_9O_2N$. MW, 175. Needles from EtOH. M.p. 78–9°. B.p. 285°/758 mm.

N-2-*Chloroethyl*: $C_{10}H_8O_2NCl$. MW, 209·5. M.p. 79–81°.

N-2-*Bromoethyl*: $C_{10}H_8O_2NBr$. MW, 254. M.p. 82–83·5°.

N-2-*Iodoethyl*: $C_{10}H_8O_2NI$. MW, 301. M.p. 99–100°.

N-*Propyl*: $C_{11}H_{11}O_2N$. MW, 189. M.p. 66°. B.p. 296·9°/758 mm. (282–3°/756 mm.).

N-2-*Chloropropyl*: $C_{11}H_{10}O_2NCl$. MW, 223·5. M.p. 100–2°.

N-3-*Chloropropyl*: m.p. 67–8°.

N-2 : 3-*Dichloropropyl*: $C_{11}H_9O_2NCl_2$. MW, 258. M.p. 93°.

N-2-*Bromopropyl*: $C_{11}H_{10}O_2NBr$. MW, 268. M.p. 110–11° (105°).

N-3-*Bromopropyl*: m.p. 72–3°.

N-2 : 3-*Dibromopropyl*: $C_{11}H_9O_2NBr_2$. MW, 347. M.p. 113–14°.

N-3-*Iodopropyl*: $C_{11}H_{10}O_2NI$. MW, 315. M.p. 88°.

N-*Isopropyl*: m.p. 85°. B.p. 286°/761 mm.

N-2-*Chloroisopropyl*: m.p. 56–8°.

N-2-*Bromoisopropyl*: m.p. 59–60°.

N-*Butyl*: $C_{12}H_{13}O_2N$. MW, 203. B.p. 311–18°/758 mm.

N-4-*Bromobutyl*: $C_{12}H_{12}O_2NBr$. MW, 282. M.p. 80·5°.

N-4-*Iodobutyl*: $C_{12}H_{12}O_2NI$. MW, 329. M.p. 88–89·5°.

N-*Isobutyl*: m.p. 93°. B.p. 293–5°.

N-*Amyl*: $C_{13}H_{15}O_2N$. MW, 217. M.p. 23°. B.p. 303°. D_4^{25} 1·093. $[\alpha]_D^{25} + 7·53°$.

N-*Isoamyl*: m.p. 12·5°. B.p. 307–8°.

N-*Octyl*: $C_{16}H_{21}O_2N$. MW, 259. M.p. 48–9°. B.p. about 216°/20 mm.

N-*Cyclopropyl*: $C_{11}H_9O_2N$. MW, 187. M.p. 135–6°.

N-α-*Camphyl*: $C_{18}H_{21}O_2N$. MW, 283. M.p. 54°.

N-*Vinyl*: $C_{10}H_7O_2N$. MW, 173. M.p. 86°.

N-*Propenyl*: $C_{11}H_9O_2N$. MW, 187. Yellow leaflets from EtOH. M.p. 151°.

N-*Isopropenyl*: m.p. 105–6°.

N-*Allyl*: $C_{11}H_9O_2N$. MW, 187. Cryst. from EtOH. M.p. 71°. B.p. 295°.

N-*Phenyl*: *see* Phthalanil.

N-p-*Chlorophenyl*: $C_{14}H_8O_2NCl$. MW, 257·5. M.p. 194–5°.

N-2 : 4-*Dichlorophenyl*: $C_{14}H_7O_2NCl_2$. MW, 292. M.p. 155°.

N-p-*Bromophenyl*: $C_{14}H_8O_2NBr$. MW, 302. M.p. 203–4°.

N-2 : 4-*Dibromophenyl*: $C_{14}H_7O_2NBr_2$. MW, 381. M.p. 153·5°.

N-p-*Iodophenyl*: $C_{14}H_8O_2NI$. MW, 349. M.p. 227–8° (235°).

N-o-*Nitrophenyl*: $C_{14}H_8O_4N_2$. MW, 268. M.p. 202–3°.

N-m-*Nitrophenyl*: m.p. 242–4°.

N-p-*Nitrophenyl*: m.p. 264–6°.

N-*Picryl*: $C_{14}H_6O_8N_4$. MW, 358. M.p. 259°.

N-*Benzyl*: $C_{15}H_{11}O_2N$. MW, 237. M.p. 115–16°.

N-o-*Tolyl*: $C_{15}H_{11}O_2N$. MW, 237. M.p. 182°.

N-m-*Tolyl*: m.p. 170–2°.

N-p-*Tolyl*: m.p. 204°.

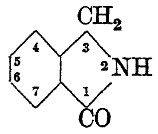
N-*Cinnamyl*: $C_{17}H_{13}O_2N$. MW, 263. M.p. 153°.

N-1-*Naphthyl*: $C_{18}H_{11}O_2N$. MW, 273. M.p. 180–1°.

N-2-*Naphthyl*: m.p. 216°.

Herzog, *Z. angew. Chem.*, 1919, **32**, 301.
Tseng, *Chem. Abstracts*, 1934, **28**, 6133.
Inone, Horiguchi, *Chem. Abstracts*, 1933, **27**, 2684.
Jaeger, Daniels, U.S.P., 1,966,068, (*Chem. Abstracts*, 1934, **28**, 5476); U.S.P., 1,914,723, (*Chem. Abstracts*, 1933, **27**, 4243); U.S.P., 1,968,253, (*Chem. Abstracts*, 1934, **28**, 5835).
Manske, *Organic Syntheses*, 1932, XII, 10.
Salzberg, Supniewski, *Organic Syntheses*, 1927, VII, 8.

Phthalimidine (1-*Isoindolenone*, *benzylamine*-o-*carboxylic lactam*)



C_8H_7ON MW, 133

Needles from H_2O. M.p. 150° (149°). B.p. 336–7°/730 mm. Sol. EtOH, Et_2O, $CHCl_3$.

B,HCl: m.p. 150° decomp.

N-*Acetyl*: m.p. 151°.

N-*Me*: C_9H_9ON. MW, 147. M.p. 120°. B.p. 300°.

N-*Et*: $C_{10}H_{11}ON$. MW, 161. M.p. 45°. B_2,H_2PtCl_6: m.p. 145°.

N-*Isobutyl*: $C_{12}H_{15}ON$. MW, 189. B.p. 310–12°/740 mm.

N-*Phenyl*: *see N*-Phenylphthalimidine.

N-*Benzyl*: $C_{15}H_{13}ON$. MW, 223. M.p. 90–1°.

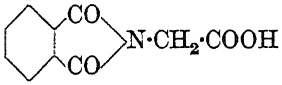
N-o-*Hydroxybenzyl*: $C_{15}H_{13}O_2N$. MW, 239. M.p. 159–60°.

N-p-*Hydroxybenzyl*: red needles from H_2O. M.p. 187–98°.

N-o-*Aminobenzyl*: $C_{15}H_{14}ON_2$. MW, 238. M.p. 153–4°.
N-p-*Aminobenzyl*: m.p. 187–8°.
N-*Nitroso*: m.p. 150°.
Picrate: m.p. 140°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, **13**, 469.
Sakurai, *Bull. Chem. Soc. Japan*, 1930, **5**, 184.
Packendorff, *Ber.*, 1934, **67**, 907.

Phthaliminoacetic Acid (*Phthaloylglycine*)

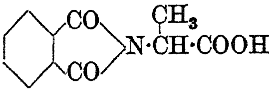


$C_{10}H_7O_4N$ MW, 205

Prisms or needles. From H_2O. M.p. 192–3°. Sol. hot H_2O, hot EtOH. Insol. $CHCl_3$, pet. ether, ligroin. $k = 1 \times 10^{-3}$ at 25°.
Et ester: $C_{12}H_{11}O_4N$. MW, 233. Needles from H_2O. M.p. 104–5° (112–13°).
Chloride: $C_{10}H_5O_3NCl$. MW, 222·5. Needles from ligroin. M.p. 84–5°.
Anhydride: $C_{20}H_{12}O_7N_2$. MW, 392. Needles from $PhNO_2$. M.p. 242°.
Anilide: m.p. 231–2°.
Phenylhydrazide: m.p. 199°.

Johnson, Scott, *J. Am. Chem. Soc.*, 1913, **35**, 1133.
Reise, *Ann.*, 1887, **242**, 1.
Gabriel, *Ber.*, 1907, **40**, 2649.
Scheiber, *Ber.*, 1913, **46**, 1103.
Billman, Harting, *J. Am. Chem. Soc.*, 1948, **70**, 1473.
Wanag, Veinbergs, *Ber.*, 1942, **75**, 1558.

1-Phthaliminopropionic Acid (N-*Phthaloylalanine*)



$C_{11}H_9O_4N$ MW, 219

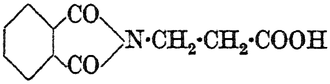
d-.
Et ester: $C_{13}H_{13}O_4N$. MW, 247. Cryst. from ligroin. M.p. 58–61°.

l-.
M.p. 150–1°. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. ligroin. $[\alpha]_D^{20} - 17·8°$ in EtOH.
Et ester: cryst. from ligroin. M.p. 54–6°. $[\alpha]_D^{20} - 12·5°$ in EtOH.

dl-.
Needles from H_2O. M.p. 160–2° (164°). Sol. EtOH, Et_2O, Me_2CO, AcOH, hot H_2O. Insol. pet. ether.
Et ester: plates from CS_2. M.p. 65° (61–3°).
Phenyl ester: $C_{17}H_{13}O_4N$. MW, 295. Needles from EtOH. M.p. 99°. Sol. Et_2O, C_6H_6, AcOH, hot EtOH.
Chloride: $C_{11}H_8O_3NCl$. MW, 237·5. Cryst. from ligroin. M.p. 73° (71°).

Bayer, D.R.P., 209,962, (*Chem. Zentr.*, 1909, I, 1951).
Bachstez, *Ber.*, 1914, **47**, 3166.
Fischer, *Ber.*, 1907, **40**, 498.
Gabriel, *Ber.*, 1905, **38**, 634.
Billman, Harting, *J. Am. Chem. Soc.*, 1948, **70**, 1473.
Wanag, Veinbergs, *Ber.*, 1942, **75**, 1558.

2-Phthaliminopropionic Acid

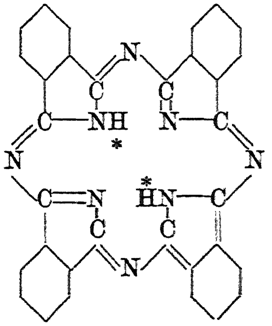


$C_{11}H_9O_4N$ MW, 219

Cryst. from H_2O. M.p. 150–1°. Sol. Et_2O, Me_2CO, AcOH, AcOEt. Spar. sol. H_2O, EtOH, C_6H_6. Insol. ligroin.
Me ester: cryst. from EtOH–C_6H_6. M.p. 73–5°.
Et ester: needles. M.p. 73·5°.
Isoamyl ester: $C_{16}H_{19}O_4N$. MW, 289. M.p. 61°.
Chloride: m.p. 107–8°.

Hale, Britton, *J. Am. Chem. Soc.*, 1919, **41**, 845.
Chodroff, Kapp, Beckmann, *J. Am. Chem. Soc.*, 1947, **69**, 256.

Phthalocyanine

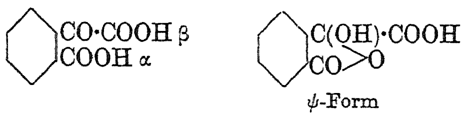


$C_{32}H_{18}N_8$ MW, 514

Greenish-blue needles with purple reflex from quinoline. Sublimes at 550° decomp. under red. press. Sol. benzophenone, naphthalene, cyclohexanol, menthol, quinoline ⟶ blue sols. Sol. aniline and homologues ⟶ green sols. Very stable. Forms metallic derivs. at *N* atoms marked *, of formula $(C_8H_4N_2)_4M$, very stable blue cryst. solids.

Robertson, *J. Chem. Soc.*, 1935, 615.
Linstead, *J. Chem. Soc.*, 1934, 1020, 1035.
Barnett, Dent, Linstead, *J. Chem. Soc.*, 1936, 1719.
Linstead, Robertson, *ibid.*, 1736.
Linstead, Cook, *J. Chem. Soc.*, 1938, 1151.

Phthalonic Acid (o-*Carboxybenzoylformic acid*)



ψ-Form

$C_9H_6O_5$ MW, 194

Cryst. + $2H_2O$ from H_2O. M.p. anhyd. 146°. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. C_6H_6, $CHCl_3$, pet. ether. Ox. ⟶ phthalic acid.

α-Me ester: $C_{10}H_8O_5$. MW, 208. Cryst. + $1H_2O$. M.p. 79–81°. $k = 1{\cdot}5 \times 10^{-4}$ at 25°.

Di-Me ester: $C_{11}H_{10}O_5$. MW, 222. Cryst. from MeOH. M.p. 66–8°.

α-Amide: α-phthalonamic acid. $C_9H_7O_4N$. MW, 193. Prisms + $1\frac{1}{2}H_2O$ from H_2O. M.p. anhyd. 178–9° decomp.

β-Amide: β-phthalonamic acid. M.p. 155° decomp. Sol. H_2O.

Anhydride: $C_9H_4O_4$. MW, 176. Needles from Ac_2O. M.p. 185–6° (190–1°) decomp. (rapid heat.). Spar. sol. Et_2O.

Anilide: phthalonanilic acid. $C_{15}H_{11}O_4N$. MW, 269. Needles from $CHCl_3$. M.p. 176°.

Dianilide: phthalonanilide. $C_{21}H_{16}O_3N_2$. MW, 344. Needles from EtOH. M.p. 206–8°.

ψ-Acetyl: m.p. 185–6° ⟶ phthalic anhydride + acetic acid.

ψ-Chloride: $C_9H_5O_4Cl$. MW, 212·5. M.p. 133°.

ψ-Dichloride: $C_9H_4O_3Cl_2$. MW, 231. M.p. 70°. B.p. 120°/12 mm.

ψ-Phenylhydrazone: m.p. 171–2°.

Tscherniac, *J. Chem. Soc.*, 1916, **109**, 1236 (*Bibl.*).
Cornillot, *Ann. Chim.*, 1927, **7**, 227; *Compt. rend.*, 1924, **179**, 274.
Kuroda, Perkin, *J. Chem. Soc.*, 1923, **123**, 2106.
Fuson, *J. Am. Chem. Soc.*, 1926, **48**, 1093.
Gabriel, Colman, *Ber.*, 1900, **33**, 999.

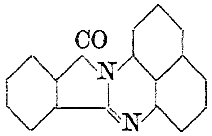
Phthalonitrile (*Phthalic acid dinitrile*, o-*dicyanobenzene*)

$C_8H_4N_2$ MW, 128

Needles from H_2O. M.p. 141° (140°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O, ligroin. Volatile in steam. Slowly hyd. by hot HCl to the acid.

Braun, Tscherniac, *Ber.*, 1907, **40**, 2710.
I.C.I., B.P. 413,639, (*Chem. Abstracts*, 1935, **29**, 178).
S.C.I., F.P. 844,567, (*Chem. Abstracts*, 1940, **34**, 7299).

10-Phthaloperinone

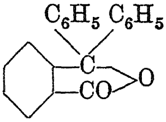


$C_{18}H_{10}ON_2$ MW, 270

Red cryst. from AcOH. M.p. 232°.

Wanay, *Ber.*, 1942, **75**, 719.

Phthalophenone (*Diphenylphthalide*, *triphenylcarbinol*-o-*carboxylic lactone*)

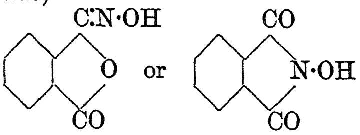


$C_{20}H_{14}O_2$ MW, 286

Leaflets from EtOH. M.p. 116°. B.p. 235°/15 mm. H_2SO_4 ⟶ greenish-yellow col. ⟶ violet on heating.

Heller, *Ber.*, 1912, **45**, 667.
Copisarow, *J. Chem. Soc.*, 1917, **111**, 18.
Scheiber, *Ann.*, 1912, **389**, 124.
Fuson, Speck, *J. Org. Chem.*, 1945, **10**, 55.

Phthaloxime (*Isonitrosophthalide*, N-*hydroxyphthalimide*)



$C_8H_5O_3N$ MW, 163

(i) Needles from EtOH. M.p. 220–6° decomp. (rapid heat. in sealed tube). Sol. hot EtOH, hot Me_2CO. Mod. sol. hot H_2O. Insol. Et_2O, C_6H_6.

Acetyl deriv.: m.p. 183–5°.

Benzoyl deriv.: m.p. 171·5°.

O-*Me*: $C_9H_7O_3N$. MW, 177. Needles from EtOH. M.p. 133°.

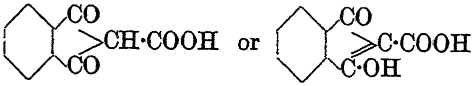
O-*Et*: $C_{10}H_9O_3N$. MW, 191. Prisms. M.p. 95–100°.

(ii) Yellow needles from EtOH. M.p. 220–6° decomp. (rapid heat. in sealed tube).

Acetyl and benzoyl derivs., *O*-Me and *O*-Et ethers identical with corresponding derivs. of (i).

Basler Chem. Fabrik, D.R.P., 130,681, (*Chem. Zentr.*, 1902, I, 1184).
Brady, Baker, Goldstein, Harris, *J. Chem. Soc.*, 1928, 538.

Phthaloylacetic Acid (1 : 3-*Diketohydrindene-2-carboxylic acid*)



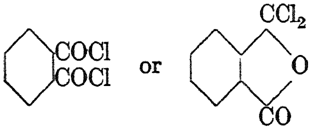
$C_{10}H_6O_4$ MW, 190

M.p. 270° decomp. Unstable, readily losing $CO_2 \longrightarrow$ 1 : 3-diketo-hydrindene.

Et ester: $C_{12}H_{10}O_4$. MW, 218. Yellow needles. M.p. 75–8°. Sol. EtOH, Et_2O, C_6H_6, ligroin. Insol. H_2O. Sol. alkalis $\longrightarrow$ enol salts. $FeCl_3 \longrightarrow$ red col. with EtOH sol.

Gabriel, Neumann, *Ber.*, 1893, **26**, 953.
Yale, *J. Am. Chem. Soc.*, 1947, **69**, 1547.

Phthaloyl chloride (3 : 3-*Dichlorophthalide*)



I. II.

$C_8H_4O_2Cl_2$ MW, 203

I.

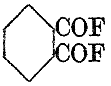
F.p. 12°. M.p. 15–16°. B.p. 281·1°, 275·4°/726 mm., 47°/130 mm. D_4^{20} 1·4089. $n_D^{15\cdot5}$ 1·517. Heat of comb. C_p 802·03 Cal., C_v 802·05 Cal.

II.

M.p. 88–9°. B.p. 275·2°/719·8 mm.

v. Braun, Kaiser, *Ber.*, 1922, **55**, 1305.
Ott, *Organic Syntheses*, 1931, XI, 88.
Garner, Sugden, *J. Chem. Soc.*, 1927, 2877.
Monsanto, B.P. 418,162, (*Chem. Abstracts*, 1935, **29**, 1436); U.S.P., 1,906,761, (*Chem. Abstracts*, 1933, **27**, 3484).
Ott, Langenohl, Zerweck, *Ber.*, 1937, **70**, 2360.

Phthaloyl fluoride



$C_8H_4O_2F_2$ MW, 170

Plates from pet. ether. M.p. 42–3°. B.p. 224–6°/760 mm., 142°/65 mm., 135°/15 mm. Lachrymatory. $H_2O \longrightarrow$ phthalic anhydride.

Dunn, Davies, Hambly, Paul, Semmens, *J. Chem. Soc.*, 1933, 15.

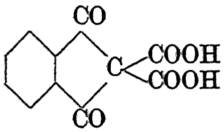
Phthaloylglycine.

See Phthaliminoacetic Acid.

Phthaloylhydrazine.

See 1 : 4-Phthalazinedione.

Phthaloylmalonic Acid (1 : 3-*Diketohydrindene*-2 : 2-*dicarboxylic acid*)



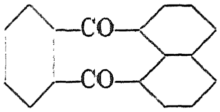
$C_{11}H_6O_6$ MW, 234

Di-Et ester: $C_{15}H_{14}O_6$. MW, 290. M.p. 74–5°. Sol. Et_2O, hot EtOH. $D_4^{83\cdot5}$ 1·1896. $n_D^{84\cdot4}$ 1·541.

Et ester-nitrile: $C_{13}H_9O_4N$. MW, 243. (i) M.p. 190–2°. Spar. sol. $CHCl_3$, C_6H_6. (ii) M.p. 140–1°.

Scheiber, Hofer, *Ber.*, 1920, **53**, 898.

1 : 8-Phthaloylnaphthalene



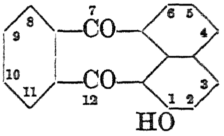
$C_{18}H_{10}O_2$ MW, 258

Needles from EtOH. M.p. 178°. $H_2SO_4 \longrightarrow$ yellow col. with green fluor.

Mono-dinitrophenylhydrazone: m.p. 265°.

Rieche, Sauthoff, Müller, *Ber.*, 1932, **65**, 1380.

1 : 8-Phthaloyl-2-naphthol (1-*Hydroxy*-7 : 12-*dihydropleiadenedione*-7 : 12)



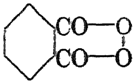
$C_{18}H_{10}O_3$ MW, 274

Yellowish-green needles from chlorobenzene. M.p. 196°. Forms no phenylhydrazone.

Acetyl deriv.: m.p. 216°.
Benzoyl deriv.: m.p. 213°.
7-*Oxime*: m.p. 242°. *Acetyl deriv.*: m.p. 237°.
12-*Oxime*: m.p. 262°. *Acetyl deriv.*: m.p. 155–60°.
Me ether: $C_{19}H_{12}O_3$. MW, 288. M.p. 205°.
Et ether: $C_{20}H_{14}O_3$. MW, 302. Yellow leaflets from Et_2O. M.p. 163·5°.

Riche, Sauthoff, Müller, *Ber.*, 1932, **65**, 1379.

Phthaloyl peroxide

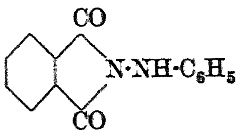


$C_8H_4O_4$ MW, 164

Cryst. M.p. 133·5° decomp. Explodes at 136°. Insol. ord. org. solvents.

Pechmann, Vanino, *Ber.*, 1894, **27**, 1511.
Baeyer, Villiger, *Ber.*, 1901, **34**, 762.

Phthaloylphenylhydrazine (N-*Anilinophthalimide*)



$C_{14}H_{10}O_2N_2$ MW, 238

(i) Yellow prisms from Me_2CO. M.p. 184°. D_4^{15} 1·356.
(ii) Plates from Me_2CO. Does not melt. D_4^{17} 1·354. Heat $\longrightarrow$ (i).

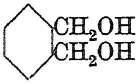
Sol. $CHCl_3$, Me_2CO, hot EtOH, hot. AcOH. Spar. sol. Et_2O, C_6H_6. Insol. H_2O, ligroin.

Scheiber, *Ann.*, 1912, **389**, 152.

Phthaloylpyridine.
See Anthrapyridinequinone.

Phthaloylquinoline.
See Anthraquinolinequinone.

Phthalyl Alcohol (o-*Xylylene glycol*, α:α-o-*xylenediol*, 1 : 2-*di*-(*hydroxymethyl*)-*benzene*, ωω′-*dihydroxy*-o-*xylene*)



$C_8H_{10}O_2$ MW, 138

Plates from Et_2O. M.p. 64·2–64·8° (62·5°). Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6.

Diacetyl: m.p. 37°.

Di-Et ether: $C_{12}H_{18}O_2$. MW, 194. Oil. B.p. 246–8°/738 mm.

Perkin, *J. Chem. Soc.*, 1888, **53**, 7.
Wislicenus, Penndorf, *Ber.*, 1910, **43**, 1837.
Nystrom, Brown, *J. Am. Chem. Soc.*, 1947, **69**, 1198.

Phthiocerane

$C_{34}H_{70}(C_{35}H_{72})$ MW, 478 (492)

Cryst. from AcOEt. M.p. 58·5–59·5°.

Stodola, Anderson, *J. Biol. Chem.*, 1936, **114**, 467.

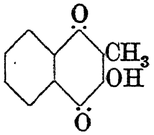
Phthiocerol

$C_{35}H_{72}O_3(C_{36}H_{74}O_3)$ MW, 540 (554)

Occurs in the wax from human and bovine tubercle bacilli. Prismatic needles from AcOEt or Me_2CO. M.p. 73–4°. $[\alpha]_D$ − 4·8° in $CHCl_3$.

Stodola, Anderson, *J. Biol. Chem.*, 1936, **114**, 467.
Cason, Anderson, *J. Biol. Chem.*, 1937, **119**, 549.
Ställberg-Stenhagen, Stenhagen, *J. Biol. Chem.*, 1948, **173**, 383.

Phthiocol (3-*Hydroxy*-2-*methyl*-1 : 4-*naphthoquinone*)



$C_{11}H_8O_3$ MW, 188

Pigment from tubercle bacilli. Possesses vitamin K activity. Yellow prisms from MeOH.Aq. M.p. 173–4°. Volatile in steam. Sol. dil. alkalis with intense red col. Ox. ⟶ phthalic acid. Zn + AcOH + AcONa ⟶ 1 : 3 : 4-triacetoxy-2-methylnaphthalene.

Acetyl deriv.: yellowish needles from MeOH. M.p. 108° (107–8°). Sol. alkalis to bright red sols.

Benzoyl deriv.: m.p. 129–130·5°.

Monoxime: m.p. 199–200°.

Monophenylhydrazone: m.p. 182–3°.

Di-nitrophenylhydrazone: does not melt below 270°.

Me ether: $C_{12}H_{10}O_3$. MW, 202. M.p. 93°.

Fieser, *J. Biol. Chem.*, 1940, **133**, 392.
Soliman, Latif, *J. Chem. Soc.*, 1944, 55.
Fieser, Oxford, *J. Am. Chem. Soc.*, 1942, **64**, 2064.
Asano, Hase, *Chem. Abstracts*, 1942, **36**, 2259.

Phthioic Acid.

The name given to acidic material of fatty acid type obtained from tubercle bacilli. The material was originally thought to be homogeneous but has now been shown to be a complex mixture of acids of which one, mycolipenic acid-I (*q.v.*), has so far been obtained pure.

Spielman, Anderson, *J. Biol. Chem.*, 1935–36, **112**, 759.
Chanley, Polgar, *Nature*, 1950, **166**, 693.
Cason, Sumrell, *J. Am. Chem. Soc.*, 1950, **72**, 4837.
Polgar, Robinson, *Chemistry and Industry*, 1951, 685.

Phyllocladene

$C_{20}H_{32}$ MW, 272

Found in the leaf oils of *Phyllocladus alpinus*, *Phyllocladus rhomboidalis* and *Araucaria excelsa*. Plates from EtOH or MeOH. M.p. 98°. $[\alpha]_D^{25}$ + 15·8° in $CHCl_3$. Isomerised by H_2SO_4 in EtOH to isophyllocladene.

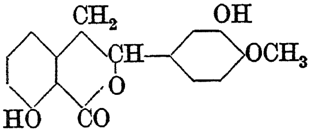
Hydrochloride: plates. M.p. 105–7° decomp.

Nitrosochloride: plates from Me_2CO–$CHCl_3$. M.p. 128° decomp.

Nitrosate: prisms from Me_2CO–$CHCl_3$. M.p. 129·5° decomp.

Briggs, *J. Soc. Chem. Ind.*, 1937, **56**, 137; *J. Chem. Soc.*, 1937, 79.

Phyllodulcin (8 : 3′-*Dihydroxy*-4′-*methoxy*-3-*phenyl*-3 : 4-*dihydroisocoumarin*)



$C_{16}H_{14}O_5$ MW, 286

d-.

Occurs in leaves of *Hydrangea thumbergii*, Sieb. M.p. 120°. $[\alpha]_D$ + 67·7 to 69·7°. It was originally thought that a natural preparation consisted of a mixture of *d*-phyllodulcin + another compound which was given the name *d*-isophyllodulcin. The mixture has now been shown to consist of *d*- + *dl*-phyllodulcin and accordingly isophyllodulcin does not exist.

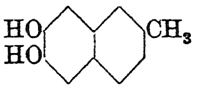
Diacetyl deriv.: m.p. 148–9°.

Dibenzoyl deriv.: m.p. 183–183·5°.

dl-.
M.p. 131°.
Di-Me ether: $C_{18}H_{18}O_5$. MW, 314. (i) Needles from EtOH. M.p. 105°. Labile. (ii) M.p. 125°. Stable.

Maniwa, *Chem. Abstracts*, 1924, **18**, 2694.
Asahina, Asano, *Ber.*, 1931, **64**, 1253.

Phyllomerol (6 : 7-*Dihydroxy*-2-*methylnaphthalene*)

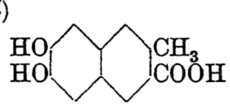


$C_{11}H_{10}O_2$ MW, 174

Cryst. M.p. 161–2°. $FeCl_3$ ⟶ green col. in MeOH.
Me ether: $C_{12}H_{12}O_2$. MW, 188. M.p. 119–22°.
Di-Me ether: $C_{13}H_{14}O_2$. MW, 202. Needles. M.p. 98–100°. *Picrate*: m.p. 120–1°.
Methylene ether: *see* Podophyllomerol.

Borsche, Niemann, *Ber.*, 1932, **65**, 1633; *Ann.*, 1933, **502**, 268.

Phyllomeronic Acid (6 : 7-*Dihydroxy*-2-*methylnaphthalene*-3-*carboxylic acid, phyllomerol*-3-*carboxylic acid*, 6 : 7-*dihydroxy*-3-*methyl*-2-*naphthoic acid*)

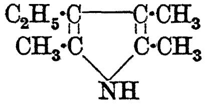


$C_{12}H_{10}O_4$ MW, 218

Leaflets from MeOH. M.p. 243–4°. $FeCl_3$ ⟶ deep blue col.
6 : 7-*Diacetyl*: m.p. 220°.
Me ester: $C_{13}H_{12}O_4$. MW, 232. Needles from MeOH. M.p. 186–7°. $FeCl_3$ ⟶ green col.
Di-Me ether: $C_{14}H_{14}O_4$. MW, 246. Needles from MeOH. M.p. 223–5°. *Me ester*: $C_{15}H_{16}O_4$. MW, 260. Needles from MeOH. M.p. 125–6°.
Methylene ether: *see* Podophyllomeronic Acid.

Borsche, Niemann, *Ber.*, 1932, **65**, 1633; *Ann.*, 1932, **499**, 68.

Phyllopyrrole (2 : 3 : 5-*Trimethyl*-4-*ethylpyrrole*)

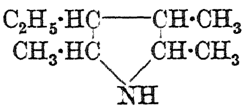


$C_9H_{15}N$ MW, 137

Leaflets from Et_2O. M.p. 66–7°. B.p. 213°/725 mm., 110–12°/35 mm., 92–3°/12 mm. Turns oily then resinous in air.
Picrate: m.p. 101–3°.

Siedel, *Z. physiol Chem.*, 1935, **231**, 167.
Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 712.
Colacicchi, *Atti accad. Lincei*, 1913, **21**, i, 489.

Phyllopyrrolidine (*Tetrahydrophyllopyrrole*)

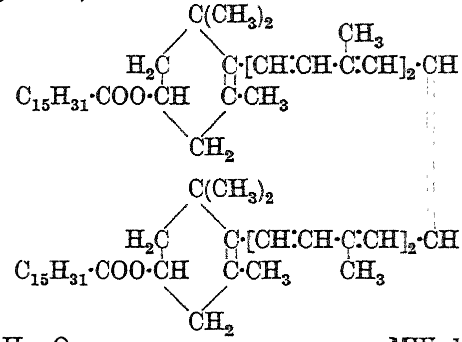


$C_9H_{19}N$ MW, 141

B.p. 160–4°. D_4^{20} 0·824.
1-*Naphthylurea*: m.p. 145°.

Willstätter, Asahina, *Ann.*, 1911, **385**, 215.

Physalien (*Zeaxanthin dipalmitate, physalin, physaliene*)



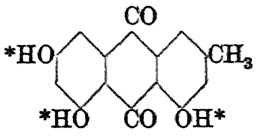
$C_{72}H_{116}O_4$ MW, 1044

Occurs in berries of *Physalis* species. Red cryst. from C_6H_6–MeOH. M.p. 98·5–99·5° (95°). $[\alpha]_c$ − 45° in $CHCl_3$, − 31° in C_6H_6. Isomerised at room temp., or more readily in boiling C_6H_6 or C_6H_6 + I ⟶ neophysalien.

Winterstein, Ehrenberg, *Z. physiol. Chem.*, 1932, **207**, 25.
Kuhn, Grundmann, *Ber.*, 1934, **67**, 596.
Karrer, Solmssen, Walker, *Helv. Chim. Acta*, 1934, **17**, 417.
Cholnoky, *Z. physiol. Chem.*, 1930, **189**, 159.
Kylin, *Z. physiol. Chem.*, 1927, **163**, 229.
Kuhn, Wiegand, *Helv. Chim. Acta*, 1929, **12**, 499.
Zechmeister, Cholnoky, Polgar, *Ber.*, 1939, **72**, 1678.
Karrer, Gugelmann, *Helv. Chim. Acta*, 1937, **20**, 405.

Physciaic Acid.
See Physcione.

Physcione (*Physciaic acid, parietin, chrysophyscin, rheumemodin methyl ether, frangulaemodin methyl ether, rheochrysidine*)



$C_{16}H_{12}O_5$ MW, 284

Occurs in chrysarobin, rhubarb root and lichens. M.p. 207°. Sol. C_6H_6, $CHCl_3$, Py,

* One of these groups is OCH_3 but which one is not known.

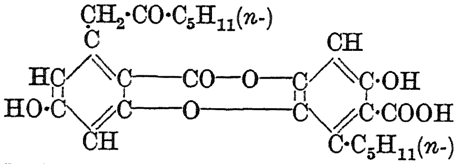
toluene. Spar. sol. AcOH, AcOEt. Insol. EtOH, MeOH, Et_2O, Me_2CO.

Tutin, Clewer, *J. Chem. Soc.*, 1912, **101**, 294.
Eder, Hauser, *Helv. Chim. Acta*, 1925, **8**, 140 (*Bibl.*).
Hesse, *Ann.*, 1917, **413**, 368.

Physeteric Acid.
See 4-Tetradecylenic Acid.

Physodalic Acid.
See Physodic Acid.

Physodic Acid (*Physodalic acid*)



$C_{26}H_{30}O_8$ MW, 470

Occurs in thalli of the lichens *Parmelia physodes* and *Parmelia vittata*, and in *Cetraria islandica*. Needles from MeOH.Aq. M.p. 205°. $FeCl_3$ ⟶ violet col. Zn + NaOH ⟶ atranol + orcinol.

Diacetyl deriv.: needles from Me_2CO.Aq. M.p. 154° (153–153·5°).
Oxime: m.p. 209–10°.
Phenylhydrazone: m.p. 265° decomp.
Me ester: $C_{27}H_{32}O_8$. MW, 484. Prisms from EtOH. M.p. 156–7°. *Me ether*: $C_{28}H_{34}O_8$. MW, 498. Leaflets from AcOH. M.p. 117–19°. *Diacetyl deriv.*: m.p. 114–15°.
Anilide: m.p. 260° decomp.

Asahina, Nogami, *Ber.*, 1935, **68**, 1500; 1934, **67**, 805.
Koller, Locker, *Monatsh.*, 1931, **58**, 209.
Fujikawa, Ishiguro, *Chem. Zentr.*, 1937, I, 2996.

Note.—Some doubt exists as to the identity of physodic with physodylic acid, *q.v.* Hesse, *J. prakt. Chem.*, 1915, **92**, 439, identifies it with capraric acid.

Physodylic Acid (*Isidic Acid*)

$C_{23}H_{24}O_6$ MW, 396

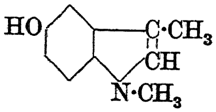
Occurs in *Evernia furfuracea*, Linn. Needles from AcOH. M.p. 196° (169–70°). Spar. sol. Et_2O.

Hesse, *J. prakt. Chem.*, 1915, **92**, 437.

Physostigmine.
See Eserine.

Physostigmine oxide.
See Geneserine.

Physostigmol (*5-Hydroxy-*1 : 3-*dimethyl-indole*)



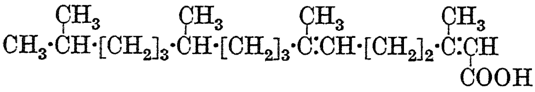
$C_{10}H_{11}ON$ MW, 161

Needles by sublimation, m.p. 114° (100°). Plates by cryst., m.p. indefinite.

Me ether: $C_{11}H_{13}ON$. MW, 175. Leaflets from MeOH. M.p. 60–1°.
Et ether: $C_{12}H_{15}ON$. MW, 189. M.p. 86° (85°). *Picrate*: m.p. 95°.

Stedman, *J. Chem. Soc.*, 1924, **125**, 1373.
Julian, Pikl, *J. Am. Chem. Soc.*, 1935, **57**, 566.
Keimatsu, Sugasawa, *Chem. Abstracts*, 1928, **22**, 3163.
Straus, *Ann.*, 1914, **406**, 337.

1 : 5-Phytadienic Acid (2 : 6 : 10 : 14-*Tetramethyl*-1 : 5-*hexadecadienoic Acid*)



$C_{20}H_{36}O_2$ MW, 308

B.p. 164°/0·25 mm.

Karrer, Koenig, *Helv. Chim. Acta*, 1941, **24**, 304.

Phytane (3 : 7 : 11 : 15-*Tetramethylhexadecane*)

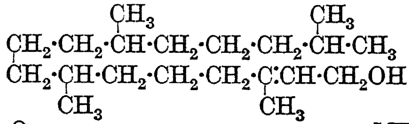
$CH_3{\cdot}CH_2{\cdot}[CH(CH_3){\cdot}CH_2{\cdot}CH_2{\cdot}CH_2]_3{\cdot}CH(CH_3)_2$

$C_{20}H_{42}$ MW, 282

B.p. 179·5°/15 mm., 169·5°/9·5 mm. Sol. pet. ether, hot EtOH, hot AcOH. D_4^0 0·803.

Karrer, *Helv. Chim. Acta*, 1929, **12**, 906.

Phytol (3 : 7 : 11 : 15-*Tetramethyl-2-hexadecenol*)



$C_{20}H_{40}O$ MW, 296

d-.

From nettles. $[\alpha]_D^{18}$ + 0·20–0·21°.
Allophanate: m.p. 71°.

l-.

Synthetic. Colourless oil. B.p. 132°/0·02 mm. $[\alpha]_D$ − 0·18°.
Allophanate: m.p. 71°.

dl-.

Decomp. product of chlorophyll. B.p. 202·5–204°/10 mm. Sol. ord. org. solvents. D_4^{25} 0·8497. n_D^{25} 1·4595.
Allophanate: m.p. 71°.
Urethane: m.p. 25·8–28·9°.
Acid phthalate: *Ag salt*, m.p. 117–19°.
Pyruvate: *semicarbazone*, needles from MeOH. M.p. 72–5°.

Fischer, Löwenberg, *Ann.*, 1929, **475**, 183.
Karrer *et al.*, *Helv. Chim. Acta*, 1943, **26**, 1741.
Smith, Sprung, *J. Am. Chem. Soc.*, 1943, **65**, 1276.
Karrer, Ringier, *Helv. Chim. Acta*, 1939, **22**, 610.
Fischer, Oestreicher, *Z. physiol. Chem.*, 1940, **262**, 243.

Phytomonic Acid

$C_{20}H_{40}O_2$ MW, 312

From the fat of *B. phytomonas tumefaciens.* M.p. 24°.

Me ester: D_4^{15} 0·8878. n^{25} 1·4500.

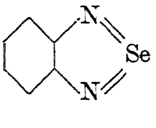
Hydrazide: m.p. 56·6°.

Velick, *J. Biol. Chem.*, 1944, **152**, 533; **156**, 101.

Phytosterol.

Generic name for the plant sterols. *See e.g.*, Fucosterol, Sitosterol *and* Stigmasterol.

Piaselenole



$C_6H_4N_2Se$ MW, 183

Fine, orthorhombic needles from H_2O, with odour of quinoline. M.p. 76°. B.p. 246°. Sol. ord. org. solvents. Spar. sol. H_2O.

B,HClO₄: canary-yellow cryst. from conc. $HClO_4$. Hyd. by H_2O.

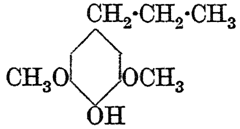
Battegay, Véchot, *Bull. soc. chim.*, 1925, **37**, 1281.

Heinemann, B.P. 3,042; D.R.P., 261,412, (*Chem. Abstracts*, 1913, **7**, 3200).

Hinsberg, *Ber.*, 1889, **22**, 2897; *J. prakt. Chem.*, 1916, **94**, 182.

Luzzati, *Compt. rend.*, 1948, **226**, 738.

Picamar (*4-Propyl-2 : 6-dimethoxyphenol, 5-propylpyrogallol-1 : 3-dimethyl ether, 4-hydroxy-3 : 5-dimethoxy-propylbenzene*)



$C_{11}H_{16}O_3$ MW, 196

Oil. B.p. 285°.

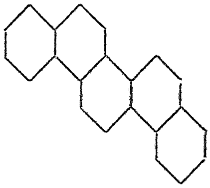
Acetyl: cryst. from EtOH. M.p. 87°. Br in CCl_4 ⟶ dibromo deriv., m.p. 101–2°.

Mauthner, *J. prakt. Chem.*, 1921, **102**, 37.

Picein.

See Piceoside.

Picene (*1 : 2 : 7 : 8-Dibenzphenanthrene, β : β-dinaphthylene-ethylene*)



$C_{22}H_{14}$ MW, 278

Colourless cryst. from Py or boiling xylene. M.p. 367–9° (366–366·5°, 365–6°). B.p. 518–20°. Sublimes at 300°/2 mm. Spar. sol. boiling C_6H_6, $CHCl_3$, AcOH. Fluoresces pale blue in ultra-violet light. Sol. conc. H_2SO_4 ⟶ intense green col.

Dibromide: m.p. 295°.

2 : 7-Dinitroanthraquinone add. comp.: m.p. 299–300°.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1934, **17**, 200, 470.

Winterstein, Schön, Vetter, *Z. physiol. Chem.*, 1934, **230**, 158.

Friedmann, *Ber.*, 1916, **49**, 277.

Meyer, Hofmann, *Monatsh.*, 1916, **37**, 715.

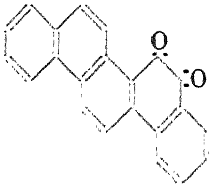
Bamberger, Chattaway, *Ann.*, 1895, **284**, 60.

Newman. *J. Org. Chem.*, 1944, **9**, 518.

Drake, McVey, *J. Org. Chem.*, 1939, **4**, 464.

Waldmann, Pitschak, *Ann.*, 1937, **527**, 183.

Picenequinone



$C_{22}H_{12}O_2$ MW, 308

Red needles or leaflets from boiling AcOH. Sublimes on slow heating. Decomp. on rapid heating. Dist. with Zn dust ⟶ picene. Dist. over lead foil in vacuo ⟶ 2′ : 1′-naphtha-1 : 2-fluorene.

Meyer, Hofmann, *Monatsh.*, 1916, **37**, 681, 721.

Cook, *J. Chem. Soc.*, 1941, 686.

Picenic Acid.

A mono-carboxylic acid derived from 2-phenyl-phenanthrene. Cryst. from EtOH or C_6H_6. M.p. 207–9° corr. Gives colour changes with H_2SO_4 characteristic of 2′ : 1′-naphtha-1 : 2-fluorenone.

Cook, *J. Chem. Soc.*, 1941, 686.

Piceoside (*Ameliaroside,* p-*hydroxyaceto-phenone-β-glucoside, picein, salinigrin*)

$C_{14}H_{18}O_7$ MW, 298

Constituent of various willow barks, *Amelanchier vulgaris*, Moench., *Picea excelsa*, Link., *Salix discolor*, Muhl, and also present in *Amelanchier* (*Rosaceae*). Cryst. + $1H_2O$ from H_2O. M.p. 195°. $[\alpha]_D$ −86·5°. Sol. EtOH, EOt_2, AcOH. Spar. sol. H_2O. Dil. acids or emulsin ⟶ *p*-hydroxyacetophenone + glucose. 1·4*N* KOH for 50 days at 20° ⟶ *p*-hydroxyaceto-phenone + β-glucosan.

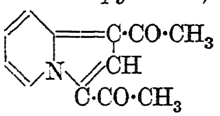
Tetra-acetyl: m.p. 172–3°.

Oxime: m.p. 228°.

Phenylhydrazone: m.p. 185°.
Semicarbazone: m.p. 220°.

Ramart-Lucas, Rabaté, *Compt. rend.*, 1933, **196**, 1493.
Jowett, *J. Chem. Soc.*, 1932, 721.
Rabaté, *Bull. soc. chim. biol.*, 1930, **12**, 146, 332, 441, 965.
Bargellini, Leone, *Atti accad. Lincei*, 1925, **2**, 35.
Mauthner, *J. prakt. Chem.*, 1913, **88**, 764.
Montgomery, Richtmyer, Hudson, *J. Am. Chem. Soc.*, 1942, **64**, 693.

Picolide (1 : 3-*Diacetoindolizine*, 1 : 3-*diacetopyrrocolin*, 1 : 3-*diacetopyrindole*)



$C_{12}H_{11}O_2N$ MW, 201

Needles from H_2O. M.p. 176°. B.p. 360° (slight decomp.). Sol. $CHCl_3$, AcOH, Py. Mod. sol. cold EtOH. Insol. Et_2O. Sol. in conc. HCl re-ppd. by H_2O. $CHCl_3$ sol. adds Br. Decolourises $KMnO_4$.Aq. Heat with 25% HCl ⟶ pyrrocolin.

Monoxime: needles from EtOH or H_2O. M.p. 244°.
Monophenylhydrazone: yellow needles from EtOH.Aq. M.p. 168°.
Monosemicarbazone: yellow plates from EtOH. M.p. 233°.

Tschitschibabin, Stepanow, *Ber.*, 1929, **62**, 1068.

α-Picoline (2-*Methylpyridine*)

C_6H_7N MW, 93

Oil with strong unpleasant odour. B.p. 129°. D_4^{15} 0·9497. n_D^{17} 1·5029. Misc. with most solvents. Heat of comb. C_v 815·4 Cal. $k = 1{\cdot}05$ (3·2) $\times 10^{-8}$ at 25°. Oxidised by boiling $KMnO_4$.Aq. or SeO_2 ⟶ picolinic acid. Reduced by Na in EtOH or by H (+ Pd or Pt) ⟶ α-pipecoline.

B,HCl: hygroscopic cryst. + $\frac{1}{2}H_2O$. M.p. anhyd. 200°.
B,HBr,Br: red cryst. M.p. 76°.
B,HBr,HgBr$_2$: needles. M.p. 88°.
B,HI,I$_2$: dark brown cryst. M.p. 44°.
B,HAuCl$_4$: cryst. M.p. 183–4° (167–8°).
B$_2$,3HgCl$_2$: needles. M.p. 170–2°.
B,HCl,2HgCl$_2$: prisms from H_2O. M.p. 154°.
B$_2$,H$_2$PtCl$_6$: cryst. M.p. 216–17° (195°) decomp.
Methiodide: needles from EtOH. M.p. 226–8°. *Chloroplatinate*: m.p. 225–6°.
Ethobromide: m.p. 97°.
Ethiodide: m.p. 123°.
Picrate: needles. M.p. 169–71° (163°).
Styphnate: m.p. 180°.

Cartwright, Errera, *Compt. rend.*, 1935, **200**, 914.
Henze, *Ber.*, 1934, **67**, 750.
Tartarini, Samaja, *Ann. chim. applicata*, 1933, **23**, 351.
Borisov, *Ber.*, 1930, **63**, 2278.
I.G., B.P. 283,163, (*Chem. Abstracts*, 1928, 22, 3892).
Wilkie, Shaw, *J. Soc. Chem. Ind.*, 1927, **46**, 469T.
Heap, Jones, Speakman, *J. Am. Chem. Soc.*, 1921, **43**, 1936.
Carter, U.S.P. 2,175,339, (*Chem. Abstracts*, 1940, **34**, 780).
Cullinane, Chard, Meatyard, *J. Soc. Chem. Ind.*, 1948, **67**, 142.

β-Picoline (3-*Methylpyridine*).

Oil with sweetish odour. B.p. 143·8°. D_4^{25} 0·9515. n_D^{24} 1·5043. Misc. with most solvents. Heat of comb. C_v 812·4 Cal. $k = 1{\cdot}1 \times 10^{-8}$ at 25°. $KMnO_4$ ⟶ nicotinic acid. Red. ⟶ β-pipecoline. $NaNH_2$ in boiling xylene ⟶ 1-amino-β-picoline, m.p. 24°. Br in CCl_4 ⟶ complex salt of bromopicoline, m.p. 200°.

B,HCl,2HgCl$_2$: cryst. from EtOH. M.p. 147–9°.
B$_2$,ZnCl$_2$: needles. M.p. 158°.
B,HAuCl$_4$: needles. M.p. 182–4°.
B$_2$,H$_2$PtCl$_6$: prisms + H_2O. M.p. anhyd. 202°.
Methiodide + *2I*: light brown leaflets from EtOH. M.p. 36°.
Picrate: m.p. 149–50°.
Styphnate: m.p. 153–4°.

Aldred, Lyons, *Chem. Abstracts*, 1931, **25**, 3345.
Henze, *Ber.*, 1934, **67**, 750.
Heap, Jones, Speakman, *J. Am. Chem. Soc.*, 1921, **43**, 1936.
Stitz, *Chem. Abstracts*, 1944, **38**, 2040.
Riethof, Richards, Savitt, Othmer, *Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 458.
Cullinane, Chard, Meatyard, *J. Soc. Chem. Ind.*, 1948, **67**, 142.

γ-Picoline (4-*Methylpyridine*).

B.p. 143·1°. D_4^{15} 0·9571. n_D^{19} 1·5064. Heat of comb. C_v 816 Cal. $k = 1{\cdot}1 \times 10^{-8}$ at 25°. $KMnO_4$ ⟶ isonicotinic acid. Red. ⟶ γ-pipecoline.

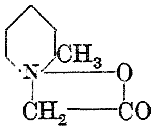
B,2HgCl$_2$: needles. M.p. 128–9°.
B,HAuCl$_4$: prisms from H_2O. M.p. 205° decomp.
B$_2$,H$_2$PtCl$_6$: leaflets. M.p. 231–44° according to rate of heating.
Methiodide: flat prisms from Me_2CO. M.p. 149–50°.
Methiodide + *2I*: light brown plates from EtOH. M.p. 101°.
Methiodide + *4I*: steel-blue needles. M.p. 63°.

Methiodide + 6*I*: dark green needles. M.p. 81·5°.

Picrate: m.p. 167°.

Bailey, McElvain, *J. Am. Chem. Soc.*, 1930, **52**, 1633.
I.G., B.P. 283,163, (*Chem. Abstracts*, 1928, **22**, 3892).
Meisenheimer, *Ann.*, 1920, **420**, 197.
Clemo, Gourlay, *J. Chem. Soc.*, 1938, 478.

α-Picoline-betaïne



$C_8H_9O_2N$ MW, 151

Needles from EtOH–Et_2O. Decomp. at 162° (turns brown above 100°). Very hygroscopic. Sol. EtOH. H_2O sol. reacts neutral.

B,HCl: m.p. 188° decomp.

B_2,H_2PtCl_6: prisms + $2H_2O$ from HCl.Aq. M.p. 212° decomp.

Kirpal, *Monatsh.*, 1910, **31**, 976.

β-Picoline-betaïne.

Leaflets + $1H_2O$ from EtOH–Et_2O. Sol. H_2O, EtOH.

B,HCl: prisms. M.p. 189° decomp.

B_2,H_2PtCl_6: plates from H_2O. Decomp. at 222°.

Krüger, *J. prakt. Chem.*, 1891, **43**, 364, 370.

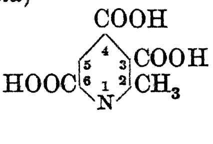
Picoline-carboxylic Acid.

See Homonicotinic Acid, Methylnicotinic Acid *and* Methylpicolinic Acid.

Picoline-dicarboxylic Acid.

See 2-Methylcinchomeronic Acid, Methyldinicotinic Acid, Methylquinolinic Acid *and* Uvitonic Acid.

α-Picoline-3 : 4 : 6-tricarboxylic Acid (*6-Methylberberonic acid, 2-methylpyridine-3 : 4 : 6-tricarboxylic acid*)



$C_9H_7O_6N$ MW, 225

Prisms or plates + $3H_2O$ from H_2O. M.p. anhyd. 226° decomp. (sealed tube). Very sol. hot H_2O. Sol. EtOH, AcOH. Spar. sol. Et_2O, C_6H_6, ligroin. On standing several days with AcOH ⟶ α-picoline-3 : 4-dicarboxylic acid. Conc. aq. sol. + ferrous ammonium sulphate ⟶ dark red col. + ppt. Warmed with Cu acetate ⟶ bluish-green ppt.

Anilide-phenylimide: $C_{21}H_{15}O_3N_3$. MW, 357. Needles. M.p. 237°.

Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, **125**, 631, 638.
Cf. Mumm, Hüneke, *Ber.*, 1918, **51**, 155.

α-Picoline-3 : 5 : 6-tricarboxylic Acid (*2-Methylpyridine-3 : 5 : 6-tricarboxylic acid, 6-methylcarbodinicotinic acid*).

Cryst. + $1H_2O$ from H_2O. Turns yellow at 170°. M.p. 226° decomp. The acid or its K salt on heating to 150° ⟶ α-picoline-3 : 5-dicarboxylic acid. $FeCl_3$.Aq. ⟶ yellow ppt.

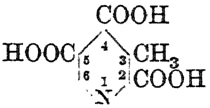
Weber, *Ann.*, 1887, **241**, 6.

α-Picoline-4 : 5 : 6-tricarboxylic Acid (*2-Methylpyridine-4 : 5 : 6-tricarboxylic acid, 6-methylcarbocinchomeronic acid*).

Prisms or plates from H_2O. Decomp. slowly on heating (turns yellow at 210°, dark brown to black at 280°). Sol. H_2O. Very spar. sol. EtOH, Et_2O. Stable to boiling AcOH. $FeSO_4$.Aq. ⟶ red col.

Mumm, Hüneke, *Ber.*, 1918, **51**, 157.

β-Picoline-2 : 4 : 5-tricarboxylic Acid (*3-Methylberberonic acid, 3-methylpyridine-2 : 4 : 5-tricarboxylic acid*)

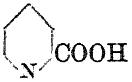


$C_9H_7O_6N$ MW, 225

Prisms + $1H_2O$. M.p. 208°. Sol. EtOH. Spar. sol. cold H_2O. Insol. Et_2O, $CHCl_3$, C_6H_6. $FeSO_4$.Aq. ⟶ weak reddish-brown col.

Dobbie, Lauder, *J. Chem. Soc.*, 1902, **81**, 151.
Dobbie, Marsden, *J. Chem. Soc.*, 1897, **71**, 663.

Picolinic Acid (*Pyridine-2-carboxylic acid*)



$C_6H_5O_2N$ MW, 123

Needles from H_2O, EtOH or C_6H_6. M.p. 136–7°. Sublimes. Very sol. AcOH. Sol. H_2O. Mod. sol. EtOH. Spar. sol. C_6H_6. Insol. Et_2O, $CHCl_3$, CS_2. Na in boiling EtOH ⟶ hexahydropicolinic acid. NaHg in H_2O ⟶ 1-hydroxyadipic acid. Conc. alc. KOH at 240° ⟶ pyridine. Zn dust + AcOH ⟶ α-picoline.

Me ester: $C_7H_7O_2N$. MW, 137. Hygroscopic cryst. M.p. 14°. B.p. 232°.

Et ester: $C_8H_9O_2N$. MW, 151. B.p. 243°, 122°/13 mm.

Phenyl ester: $C_{12}H_9O_2N$. MW, 199. M.p. 82°.

Anhydride: m.p. 124° (sealed tube).

Chloride: C_6H_4ONCl. MW, 141·5. M.p. 46°. B.p. 160° (slight decomp.)/10 mm.

Amide: $C_6H_6ON_2$. MW, 122. M.p. 106·5°.

Anilide: m.p. 76°.

p-*Toluidide*: m.p. 104°.

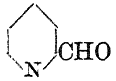
Nitrile: *see* 2-Cyanopyridine.

N-*Me-betaïne*: *see* Homarine.

Aurichloride: m.p. 200° (204° decomp.).

Platinichloride: cryst. + $2H_2O$. M.p. 215–16°.
Brode, Bremer, *J. Am. Chem. Soc.*, 1934, **56**, 993.
Hoppe-Seyler, *Z. physiol. Chem.*, 1933, **222**, 105.
Meyer, *Rec. trav. chim.*, 1925, **44**, 323.
Hess, Leibbrandt, *Ber.*, 1917, **50**, 385.
Ley, Ficken, *ibid.*, 1132.
Singer, McElvain, *Organic Syntheses*, 1940, XX, 79.
Kaslow, Stayner, *J. Am. Chem. Soc.*, 1945, **67**, 1717.

Picolinic Aldehyde (*Pyridine-2-aldehyde*)



C_6H_5ON MW, 107

Oil. B.p. 180°/750 mm., 62–3°/13–14 mm. $D_4^{18\cdot5}$ 1·1255. $n_D^{18\cdot5}$ 1·5389. Sol. H_2O, EtOH, Et_2O, AcOEt.

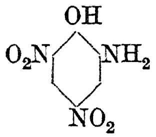
Oxime: white needles from H_2O. M.p. 113·5°.

Semicarbazone: minute needles from EtOH. M.p. 195–6°.

Phenylhydrazone, HCl: needles from EtOH. M.p. 196°.
Lénart, *Ber.*, 1914, **47**, 808.
Niemann, Lewis, Hays, *J. Am. Chem. Soc.*, 1942, **64**, 1678.

Picolylamine.
See Aminopicoline.

Picramic Acid (4 : 6-*Dinitro-2-aminophenol*)



$C_6H_5O_5N_3$ MW, 199

Dark red needles from EtOH, prisms from $CHCl_3$. M.p. 169°. Sol. C_6H_6, AcOH. Mod. sol. EtOH. Spar. sol. Et_2O, $CHCl_3$. Sol. to 0·14% in H_2O at 22°. Sol. alkalis ⟶ reddish-brown col. Colourless sol. in conc. HCl. $k = 2{\cdot}5 \times 10^{-5}$ at 0°, $1{\cdot}1 \times 10^{-4}$ at 65°. Forms salts with acids and bases. More toxic than picric acid. Intermediate for azo dyestuffs. Gives colour reactions with proteins, amino-acids and amines, but not with their salts.

N-*Acetyl*: needles from H_2O. M.p. 201°.

N-*Benzoyl*: greenish-yellow needles from AcOH or xylene. M.p. 299–30°.

N-p-*Toluenesulphonyl*: m.p. 191°. *Py salt*: m.p. 203°.

O-*Acetyl*: m.p. 193°.

O-*Benzoyl*: leaflets. M.p. 218–19°.

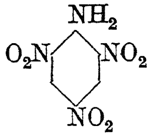
N-*Benzyl*: ochre prisms. M.p. 139–40°.

N-*Me*: orange scales from dil. EtOH. M.p. 144–5°.

N-*di-Me*: yellow micro-cryst. from AcOH. M.p. 218–20°.

Me ether: 4 : 6-dinitro-2-aminoanisole. $C_7H_7O_5N_3$. MW, 213. Violet needles from EtOH. Insol. H_2O.
Clayton, *J. Soc. Dyers Colourists*, 1930, **46**, 365, (*Chem. Abstracts*, 1931, **25**, 926).
Seyewetz, Blanc, *Chimie et Industrie*, 1930, **25**, 605.
Dehn, U.S.P., 1,472,791, (*Chem. Abstracts*, 1924, **18**, 400).
Pomeranz, *Chem.-Ztg.*, 1921, **45**, 865, (*Chem. Abstracts*, 1922, **16**, 163).
Egerer, *J. Biol. Chem.*, 1918, **35**, 565.
Aloy, Frébault, *Bull. soc. chim.*, 1904, **33**, 496.
Hofer, Jakob, *Ber.*, 1908, **41**, 3198.
Kym, *Ber.*, 1899, **32**, 1429.
Hodgson, Ward, *J. Chem. Soc.*, 1945, 663.

Picramide (2 : 4 : 6-*Trinitroaniline*)



$C_6H_4O_6N_4$ MW, 228

Prisms from AcOH. M.p. 188° (192–5°). Sol. Me_2CO, AcOEt, C_6H_6. Spar. sol. EtOH, Et_2O. Hot KOH ⟶ NH_3 + picric acid.

N-*Acetyl*: 2 : 4 : 6-trinitroacetanilide. Needles from AcOH.Aq. M.p. about 230°.

N-*Benzoyl*: 2 : 4 : 6-trinitrobenzanilide. Needles from EtOH. M.p. 195–6°.

N-*Me*: $C_7H_6O_6N_4$. MW, 242. Yellow needles from EtOH. M.p. 114·8°.

N-*Di-Me*: $C_8H_8O_6N_4$. MW, 256. Yellow plates from C_6H_6. M.p. 138°.

N-*Et*: $C_8H_8O_6N_4$. MW, 256. Cryst. from C_6H_6. M.p. 84°.

N-*Di-Et*: $C_{10}H_{12}O_6N_4$. MW, 284. Prisms from C_6H_6. M.p. 163°.

N-*Propyl*: $C_9H_{10}O_6N_4$. MW, 270. Yellow needles. M.p. 59°.

N-*Dipropyl*: $C_{12}H_{16}O_6N_4$. MW, 312. Orange-red cryst. from Me_2CO–$CHCl_3$. M.p. 38°.

N-*Isopropyl*: yellow needles from EtOH–AcOH. M.p. 106–7°.

N-*Phenyl*: *see* 2 : 4 : 6-Trinitrodiphenylamine.
Hollemann, *Rec. trav. chim.*, 1930, **49**, 112.
James, Jones, Lewis, *J. Chem. Soc.*, 1920, **117**, 1273.
Weiss, Abeles, *Monatsh.*, 1932, **59**, 238.
Spencer, Wright, *Can. J. Research*, 1946, **24*B***, 204, (*Chem. Abstracts*, 1947, **41**, 723).
Boyer, Spencer, Wright, *ibid.*, 200, (*Chem. Abstracts*, 1947, **41**, 108).

Picrasmin (*Isoquassin*)

$C_{22}H_{28}O_6$ MW, 388

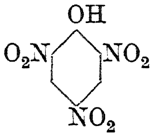
Bitter constituent of *Aeschrion excelsa* (Jamaica quassia wood). Colourless rectangular

plates and rods from MeOH.Aq. M.p. 222–5°. $[\alpha]_D^{20}$ + 46·6° in $CHCl_3$. Contains two methoxy groups.

2 : 4-*Dinitrophenylhydrazone* : fluffy orange ppt. Variable m.p.

Clark, *J. Am. Chem. Soc.*, 1938, **60**, 1146.
Adams, Whaley, *J. Am. Chem. Soc.*, 1950, **72**, 375.

Picric Acid (2 : 4 : 6-*Trinitrophenol*)



$C_6H_3O_7N_3$ MW, 229

Yellow leaflets from H_2O, prisms from Et_2O, plates from EtOH. Colourless cryst. from hot ligroin or conc. HCl. M.p. 122·5°. Sublimes with slow heat, deflagrates with rapid heat. Explosive, but salts are more so. Very bitter taste. Toxic. Polymorphic forms, m.p.s 122·5°, 101° and 75°, have been shown to exist.

Percentage solubilities :—H_2O, 0·98 at 9°, 1·18 at 20°, 1·30 at 25°, 2·33 at 50°, 7·6 at 100°; MeOH, 16 at 16°, 40·2 at 50°; EtOH, 6·8 at 16°, 19·7 at 50°; AcOEt, 39·4 at 16°, 68·5 at 50°; Me_2CO, 123·3 at 16°, 220·5 at 50°; Et_2O, 2·64 at 16°, 4·0 at 50°; $CHCl_3$, 1·86 at 20° (2·0 at 16°), 5·7 at 50°; C_6H_6, 7·5 at 16°, 29·5 at 50°; toluene, 12·2 at 16°, 27·8 at 50°; CCl_4, 0·06 at 16°, 0·07 at 20°, 0·35 at 50°; CS_2, 0·11 at 16°, 0·18 at 34°; Py, 27·62 at 16°, 58·9 at 50°. Forms add. comp. with 2Py, m.p. 144–5°. Stable to hot conc. H_2SO_4 or HCl. Red. ⟶ picramic acid ⟶ 2 : 6-diamino-4-nitrophenol ⟶ 2 : 4 : 6-triaminophenol. Cl or *aqua regia* or $KClO_3$ + HCl ⟶ chloranil + chloropicrin. PCl_5 ⟶ picryl chloride. Forms cryst. add. comps. with many bases, phenols, hydrocarbons, etc.

Molnar, *Compt. rend.*, 1935, **201**, 59.
Haid, Koenen, *Chem. Abstracts*, 1934, **28**, 2907.
I.C.I., B.P. 370,436, (*Chem. Abstracts*, 1933, **27**, 2965).
Muraour, *Bull. soc. chim.*, 1932, **51**, 1152.
Desvergnes, *Chimie et Industrie*, 1929, **22**, 451.
Benedict, *J. Biol. Chem.*, 1929, **82**, 1.
Olsen, Goldstein, *Ind. Eng. Chem.*, 1924, **16**, 66.
Robertson, Garner, *Proc. Roy. Soc.*, 1923, **103A**, 539.
Holliday, Badier, U.S.P., 1,413,914, (*Chem. Abstracts*, 1922, **16**, 2150).
Marqueyrol, Loriette, *Bull. soc. chim.*, 1919, **25**, 376.
Aristoff *et al.*, *Ind. Eng. Chem.*, 1948, **40**, 1281.

Me ether : 2 : 4 : 6-trinitroanisole. $C_7H_5O_7N_3$. MW, 243. Colourless plates or leaflets from EtOH. M.p. 68°.

Et ether: 2 : 4 : 6-trinitrophenetole. $C_8H_7O_7N_3$. MW, 257. Colourless needles from EtOH. M.p. 78·5°. Sol. Et_2O, CS_2, C_6H_6. Spar. sol. hot H_2O.

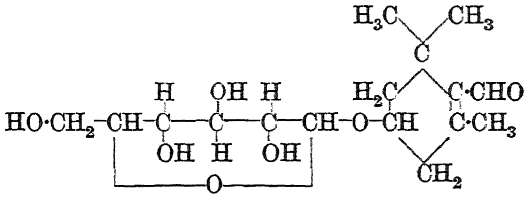
Phenyl ether : *see* 2 : 4 : 6-Trinitrodiphenyl Ether.

Benzyl ether : $C_{13}H_9O_7N_3$. MW, 319. Colourless prisms from C_6H_6. M.p. 147°.

Acetyl : colourless cryst. from Et_2O. M.p. 75–6°.

Tommasi, David, *Ann.*, 1873, **169**, 167; *Compt. rend.*, 1873, **77**, 207.
Kumpf, *Ann.*, 1884, **224**, 131.
Jackson, Gazzolo, *Am. Chem. J.*, 1900, **23**, 384.
Buttle, Hewitt, *J. Chem. Soc.*, 1909, **95**, 1759 (*Footnote*).
Jackson, Earle, *Am. Chem. J.*, 1903, **29**, 104.
Blanksma, *Chem. Zentr.*, 1909, I, 1809.
Marqueyrol, Scohy, *Bull. soc. chim.*, 1920, **27**, 105.
Guastalla, Racciu, *Industria chimica*, 1933, **8**, 1370.

Picrocrocin



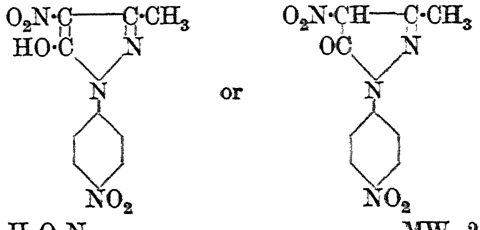
$C_{16}H_{26}O_7$ MW, 330

Terpene-glucoside, bitter principle from *Safran aquila* (*Safran electus*). Stout colourless prisms from MeOH–$CHCl_3$–Et_2O. M.p. 156°. Acid or alk. hyd. ⟶ *d*-glucose + safranal. Thought to be a female sex-determining substance (gynotermone) in *Chlamydomonas*.

Tetra-acetate : needles from pet. ether. M.p. 143°. *Semicarbazone* : m.p. 106°.

Kuhn, Winterstein, *Ber.*, 1934, **67**, 344.
Cf. Lutz, *Biochem. Z.*, 1930, **226**, 97.
Kuhn, Moewus, *Ber.*, 1940, **73**, 547.
Kuhn, Löw, *Ber.*, 1941, **74**, 219.

Picrolonic Acid (4-*Nitro*-3-*methyl*-1-p-*nitrophenylpyrazolone*-5)



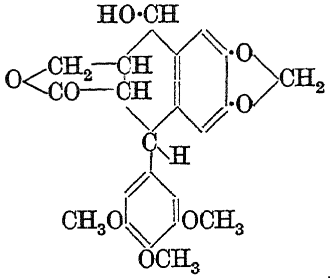
$C_{10}H_8O_5N_4$ MW, 264

Yellow cryst. M.p. 116·5° (decomp. at 125°). Sol. H_2O to 0·12% at 17°. Very spar. sol.

EtOH. Org. bases ⟶ cryst. add. comps. of definite m.p. Forms very insol. salts of Ca, Cu, Pb, hence used in detection of these metals. Deep yellow cryst. which decomp. at 220–50° have been obtained from solutions of commercial picrolonic acid.

Hugounenq, Florence, Couture, *Bull. soc. chim. biol.*, 1925, **7**, 58.
Kofler, Müller, *Chem. Abstracts*, 1937, **31**, 4667.

Picropodophyllin



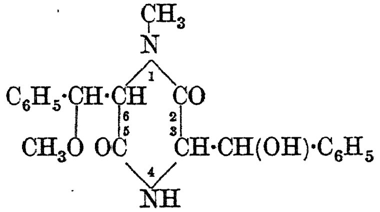
$C_{22}H_{22}O_8$ MW, 414

Constituent of purgative resin from various species of *Podophyllum.* Isomeric with podophyllotoxin but physiologically inactive. Colourless needles from MeOH or C_6H_6. Cryst. + 1 mol. solvent from MeOH or EtOH. M.p. 228°. $[\alpha]_D^{20}$ + 9·38° in $CHCl_3$. Fuming HCl at 110° in sealed tube ⟶ pyromellitic acid. Alk. $KMnO_4$ ⟶ gallic acid trimethyl ether + oxalic acid. Zn dust dist. ⟶ 1 : 6-dimethylnaphthalene.

Acetyl: $C_{24}H_{24}O_9$. MW, 456. M.p. 215–16°. $[\alpha]_D^{16}$ + 17·7° in $CHCl_3$.

Späth, Wessely, Nadler, *Ber.*, 1933, **66**, 125.
Viehoever, Mack, *J. Am. Pharm. Assocn.*, 1938, **27**, 632, (*Chem. Abstracts*, 1938, **32**, 8075).
Borsche *et al.*, *Ann.*, 1936, **526**, 1.

Picrorocellin



(An alternative structure has H at 1 and CH_3 at 4)

$C_{20}H_{22}O_4N_2$ MW, 354

Constituent of lichen *Rocella fuciformis.* Massive prisms from boiling EtOH. M.p. 190–220° according to rate of heating (192–4°). Insol. cold dil. aq. acids and alkalis. $[\alpha]_D^{18}$ + 12·5° in $CHCl_3$. Heat alone or with boiling NaOH.Aq. ⟶ anhydropicrorocellin.

O : N - *Di - Me*: dimethylpicrorocellin. $C_{22}H_{26}O_4N_2$. MW, 382. Prisms from boiling EtOH. M.p. 229°. Optically inactive. HI at 140° ⟶ 2 : 5-diketo-3 : 6-dibenzyl-1 : 6-dimethylpiperazine.

Anhydropicrorocellin: $C_{20}H_{20}O_3N_2$. MW, 336. Needles. M.p. 155°. $[\alpha]_D^{18}$ − 463·7° in $CHCl_3$. *Me ether*: prisms from EtOH–C_6H_6–pet. ether. M.p. 139°. $[\alpha]_D^{18}$ − 661·2° in $CHCl_3$.

Forster, Saville, *J. Chem. Soc.*, 1922, **121**, 816.

Picrotin

$C_{15}H_{18}O_7$ MW, 310

Di-lactone constituent of the molecular comp. picrotoxin. Colourless rods from H_2O. M.p. 255° (252°). $[\alpha]_D^{16}$ − 70° in EtOH. Alk. $KMnO_4$ ⟶ α- and β-picrotinic acids. MnO_2 + H_2SO_4.Aq. ⟶ 1 : 1-dimethylphthalide-3 : 4-dicarboxylic acid.

Mercer, Robertson, *J. Chem. Soc.*, 1936, 291; 1935, 997.
Clark, *J. Am. Chem. Soc.*, 1935, **57**, 1111.
Horrmann, *Ann.*, 1916, **411**, 273.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042, 1597, 2292.
Conroy, *J. Am. Chem. Soc.*, 1952, **74**, 491, 3046.

α-Picrotinic Acid

$C_{14}H_{19}O_6\cdot COOH$

$C_{15}H_{20}O_8$ MW, 328

Main product of prolonged hyd. of picrotin by dil. min. acids. Formed as ester together with K salt of β-picrotinic acid by action of MeOH–KOH on picrotin. Cryst. from AcOEt. M.p. 258° decomp. $[\alpha]_D^{18}$ + 71° 53′ in EtOH. Very sol. H_2O, EtOH, MeOH, Me_2CO, AcOH. Insol. $CHCl_3$, C_6H_6, ligroin. Heat in vacuo above m.p. ⟶ picrotinlactone + picrotoxic acid. 40% H_2SO_4 ⟶ picrotonol.

Me ester: $C_{16}H_{22}O_8$. MW, 342. Needles from H_2O. M.p. 239°. $[\alpha]_D^{18}$ + 77° 11′ in EtOH.

Et ester: $C_{17}H_{24}O_8$. MW, 356. Needles from H_2O. M.p. 199°. $[\alpha]_D^{18}$ + 74° 25′ in EtOH.

Horrmann, *Ann.*, 1916, **411**, 284, 298.
Horrmann, Seydel, *Ber.*, 1912, **45**, 3084.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042.

β-Picrotinic Acid

$C_{14}H_{19}O_6\cdot COOH$

$C_{15}H_{20}O_8$ MW, 328

Stout cryst. from H_2O. Decomp. at 204–5°. Sol. EtOH, MeOH, AcOH. Spar. sol. AcOEt, Me_2CO. Insol. $CHCl_3$, C_6H_6, ligroin. $[\alpha]_D^{18}$ + 4° 23′ in EtOH. Hot KOH.Aq. ⟶ picrotin-dicarboxylic acid.

K salt: cryst. from MeOH. Sinters at 245–7°, decomp. at 260°. $[\alpha]_D^{18}$ − 3° 57′ in H_2O.

Me ester: $C_{16}H_{22}O_8$. MW, 342. M.p. 231°.

Horrmann, *Ann.*, 1916, **411**, 284, 298.
Horrmann, Seydel, *Ber.*, 1912, **45**, 3084.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042.

Picrotoxic Acid

$C_{15}H_{18}O_7$ MW, 310

Needles + $2H_2O$ from H_2O. M.p. anhyd. 232°. Very sol. MeOH, EtOH, AcOH. Sol. Me_2CO, AcOEt, H_2O. Insol. C_6H_6, $CHCl_3$, ligroin. $[\alpha]_D^{18}$ + 81° 7′ in EtOH. Decolourises bromine water. H ⟶ dihydro-acid.

Me ester: $C_{16}H_{20}O_7$. MW, 324. M.p. 171·5°. $[\alpha]_D^{18}$ + 87° 32′ in EtOH.

Et ester: $C_{17}H_{22}O_7$. MW, 338. M.p. 143·5°. $[\alpha]_D^{18}$ + 81° 12′ in EtOH.

Mercer, Robertson, *J. Chem. Soc.*, 1936, 293.
Horrmann, *Ber.*, 1916, **49**, 1554; *Ann.*, 1916, **411**, 300.
Harland, Robertson, *J. Chem. Soc.*, 1939, 937.
O'Donnell, Robertson, Harland, *ibid.*, 1261.
Sutter, Schlittler, *Helv. Chim. Acta*, 1949, **32**, 1855, 1860; 1950, **33**, 902.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042, 1597, 2292.
Conroy, *J. Am. Chem. Soc.*, 1952, **74**, 491, 3046.

Picrotoxin

$C_{30}H_{34}O_{13}$ MW, 602

Bitter principle from berries of shrubs of *Anamirta cocculus*, *Menispermum cocculus*, and other species, and active principle of *Tinomiscium philippinense*. Is an equimolecular comp. of picrotin and picrotoxinin. Prisms from H_2O or EtOH. M.p. 203–4° (199–200°). $[\alpha]_D^{12}$ −30° in H_2O. Br.Aq. ⟶ monobromopicrotoxinin + picrotin. MnO_2 + H_2SO_4.Aq. ⟶ 1 : 1-dimethylphthalide-dicarboxylic acid.

Horrmann, Thilo, *Chem. Zentr.*, 1936, I, 2954.
Mercer, Robertson, *J. Chem. Soc.*, 1936, 291.
Clark, *J. Am. Chem. Soc.*, 1935, **57**, 1111.
Sielisch, *Ann.*, 1912, **391**, 1.
Harland, Robertson, *J. Chem. Soc.*, 1939, 937.
O'Donnell, Robertson, Harland, *ibid.*, 1261.
Sutter, Schlittler, *Helv. Chim. Acta*, 1949, **32**, 1855, 1860; 1950, **33**, 902.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042, 1597, 2292.
Conroy, *J. Am. Chem. Soc.*, 1952, **74**, 491, 3046.

α-Picrotoxinic Acid

$C_{14}H_{17}O_5 \cdot COOH$

$C_{15}H_{18}O_7$ MW, 310

Lactone-acid. Cubes from H_2O. M.p. 209° decomp. Very sol. MeOH, Me_2CO. Sol. EtOH, AcOEt. Insol. C_6H_6, $CHCl_3$. $[\alpha]_D^{18}$ − 5° in EtOH. Reduces cold $KMnO_4$.Aq., NH_3-$AgNO_3$ and Fehling's on warming. Decolourises bromine water. H ⟶ dihydro-deriv. Hot alkalis ⟶ picrotoxinin-dicarboxylic acid. Refluxed 15 mins. with 2*N*-H_2SO_4 ⟶ β-picrotoxinic acid.

Me ester: $C_{16}H_{20}O_7$. MW, 324. Prisms from H_2O. M.p. 182°. $[\alpha]_D^{18}$ − 9° 44′ in EtOH.

Et ester: $C_{17}H_{22}O_7$. MW, 338. Needles from H_2O. M.p. 159°. $[\alpha]_D^{18}$ − 8° 4′ in EtOH.

Horrmann, *Ber.*, 1913, **46**, 2793.
Meyer, Bruger, *Ber.*, 1898, **31**, 2958.
O'Donnell, Robertson, Harland, *J. Chem. Soc.*, 1939, 1261.
Sutter, Schlittler, *Helv. Chim. Acta*, 1950, **33**, 902.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042.
Conroy, *J. Am. Chem. Soc.*, 1952, **74**, 491.

β-Picrotoxinic Acid

$C_{14}H_{17}O_5 \cdot COOH$

$C_{15}H_{18}O_7$ MW, 310

Needles from H_2O. M.p. 235° decomp. Very sol. MeOH, EtOH, Me_2CO, AcOH. Sol. Et_2O, $CHCl_3$. Insol. C_6H_6, ligroin. $[\alpha]_D^{18}$ − 48° in EtOH. Stable in cold to Br.Aq. and $KMnO_4$.Aq. Non-reducing.

Me ester: prisms from EtOH. M.p. 204°. $[\alpha]_D^{18}$ − 50·3° in EtOH.

Et ester: prisms from H_2O. M.p. 198°. $[\alpha]_D^{18}$ − 49° 57′ in EtOH.

Horrman, *Ber.*, 1913, **46**, 2793.
Meyer, Bruger, *Ber.*, 1898, **31**, 2958.
O'Donnell, Robertson, Harland, *J. Chem. Soc.*, 1939, 1261.

Picrotoxinin

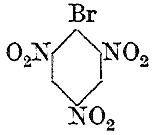
$C_{15}H_{16}O_6$ MW, 292

Di-lactone constituent of the molecular comp. picrotoxin, of which it is the physiologically active (neurophilic) component. Colourless rods from H_2O. M.p. 209·5°. Reduces $AgNO_3$ and Fehling's. Decolourises bromine water. Refluxed with 1% H_2SO_4 for 24 hours ⟶ picrotoxic acid.

Mercer, Robertson, *J. Chem. Soc.*, 1936, 291; 1935, 997.
Clark, *J. Am. Chem. Soc.*, 1935, **57**, 1111.
Harland, Robertson, *J. Chem. Soc.*, 1939, 937.
O'Donnell, Robertson, Harland, *ibid.*, 1261.
Slater, *J. Chem. Soc.*, 1943, 143.
Sutter, Schlittler, *Helv. Chim. Acta*, 1949, **32**, 1855, 1860; 1950, **33**, 902.
Slater *et al.*, *J. Chem. Soc.*, 1952, 1042, 1597, 2292.
Conroy, *J. Am. Chem. Soc.*, 1952, 491, 3046.

Picrylaniline.
See 2 : 4 : 6-Trinitrodiphenylamine.

Picryl bromide (2-*Bromo*-1 : 3 : 5-*trinitrobenzene*, 2 : 4 : 6-*trinitrobromobenzene*)

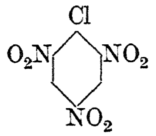


$C_6H_2O_6N_3Br$ MW, 292

Yellowish plates from EtOH–C_6H_6. M.p. 122–3°. Sol. EtOH, $CHCl_3$, AcOH, C_6H_6. Insol. H_2O. Hot NaOH.Aq. ⟶ picric acid. NaOMe ⟶ 2 : 4 : 6-trinitroanisole.

Hertel, Römer, *Z. physik. Chem.*, 1933, 22B, 267.

Picryl chloride (2-*Chloro*-1 : 3 : 5-*trinitrobenzene*, 2 : 4 : 6-*trinitrochlorobenzene*)

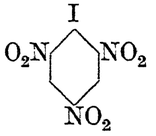


$C_6H_2O_6N_3Cl$ MW, 247·5

Needles or plates from $CHCl_3$ or EtOH–ligroin. M.p. 83°. D^{20} 1·797. Very sol. hot $CHCl_3$, C_6H_6. Sol. boiling EtOH. Spar. sol. Et_2O, hot ligroin. Insol. H_2O. Sn + HCl ⟶ 1 : 3 : 5-triaminobenzene. NH_3.Aq. ⟶ 2 : 4 : 6-trinitroaniline.

Desvergnes, *Chimie et Industrie*, 1931, **25**, 3, 291.
Hertel, Römer, *Z. physik. Chem.*, 1933, 22*B*, 267.
Boyer, Spencer, Wright, *Can. J. Research*, 1946, **24***B*, 200, (*Chem. Abstracts*, 1947, **41**, 108).

Picryl iodide (2-*Iodo*-1 : 3 : 5-*trinitrobenzene*, 2 : 4 : 6-*trinitroiodobenzene*)

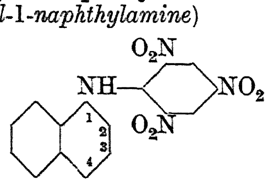


$C_6H_2O_6N_3I$ MW, 339

Golden-yellow tetragonal cryst. from C_6H_6. M.p. 164–5°. D^{22} 2·285. Hot NaOH.Aq. ⟶ picric acid.

Hertel, Römer, *Z. physik. Chem.*, 1933, 22B, 267.

***N*-Picryl-1-naphthylamine** (2 : 4 : 6-*Trinitrophenyl*-1-*naphthylamine*)



$C_{16}H_{10}O_6N_4$ MW, 354

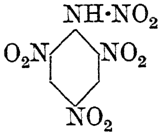
Red plates from AcOH, needles from EtOH. M.p. 198–9°. Insol. cold EtOH.
N-*Me*: $C_{17}H_{12}O_6N_4$. MW, 368. Plates from AcOH. M.p. 247° (245°). Sol. hot C_6H_6, AcOH. Spar. sol. hot EtOH.

Wedekind, *Ber.*, 1900, **33**, 435.
Busch, Kögel, *Ber.*, 1910, **43**, 1560.

***N*-Picryl-2-naphthylamine** (2 : 4 : 6-*Trinitrophenyl*-2-*naphthylamine*).
Red prisms from AcOH. M.p. 233–233·5°. Mod. sol. most org. solvents.

Bamberger, Müller, *Ber.*, 1900, **33**, 107.

Picrylnitramine (2 : 4 : 6-*Trinitrophenylnitramine*, N : 2 : 4 : 6-*tetranitroaniline*)



$C_6H_3O_8N_5$ MW, 273

Yellow cryst. from Me_2CO–$CHCl_3$. Deflagrates at 80–110°.
N-*Me*: *see* Tetryl.
N-*Et*: ethylpicrylnitramine. $C_8H_7O_8N_5$. MW, 301. Pale yellow plates from EtOH. M.p. 96°. D^{10} 1·644. Sol. $CHCl_3$, C_6H_6. Red. ⟶ 2 : 4 : 6-triaminophenol. Alkalis or NH_3 ⟶ intense red col.
N-*Propyl*: propylpicrylnitramine. $C_9H_9O_8N_5$. MW, 315. Colourless plates from EtOH. M.p. 98°. Alkalis or NH_3 ⟶ red col.
N-*Isopropyl*: isopropylpicrylnitramine. Pale yellow needles from EtOH. M.p. 108°. Sol. EtOH. D^{10} 1·563.
N-*Butyl*: butylpicrylnitramine. $C_{10}H_{11}O_8N_5$. MW, 329. Colourless plates from EtOH. M.p. 98–9°.
N-*Isobutyl*: isobutylpicrylnitramine. Needles from EtOH. M.p. 110°.

Jones, Willson, *J. Chem. Soc.*, 1930, 2277.
Duin, *Rec. trav. chim.*, 1917, **37**, 112.
Franchimont, *Rec. trav. chim.*, 1910, **29**, 300.
Romburgh, *Rec. trav. chim.*, 1885, **41**, 191.

Picylene-carbinol

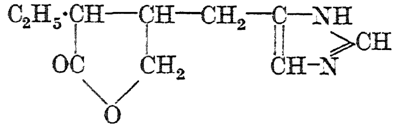
$C_{21}H_{14}O$ MW, 282

Pearly leaflets from $CHCl_3$. M.p. 230°. Sol. hot EtOH, AcOH, $CHCl_3$, C_6H_6. Spar. sol. Et_2O. Sol. in hot conc. H_2SO_4 ⟶ blue col.
Acetate: needles. M.p. 159°.

Bamberger, Chattaway, *Ann.*, 1895, **284**, 69.
Cf. Schmidlin, Hüber, *Ber.*, 1910, **43**, 2824.
Cf. Cook, *J. Chem. Soc.*, 1941, 685.

Picylene-ketone.
See 2′ : 1′-Naphtha-1 : 2-fluorenone.

Pilocarpidine



$C_{10}H_{14}O_2N_2$ MW, 194

Alkaloid occurring in leaves of *Pilocarpus Jaborandi*, Holmes. Physiological action similar to pilocarpine, but weaker.

Natural product.

Syrup. Decomp. on dist. Sol. H_2O. $[\alpha]_D^{20}$ + 81·3° in H_2O, + 35·2° in presence of alkali. Salts heated with alkali split off $NHMe_2$. NaOEt ⟶ isopilocarpidine. MeI ⟶ pilocarpine methiodide.

B,HCl: $[\alpha]_D^{20}$ + 72° in H_2O.

B,HNO₃: prisms from H_2O. M.p. 137°. $[\alpha]_D^{20}$ + 73·2° in H_2O. Mod. sol. EtOH.

B,HAuCl₄: needles from AcOH. M.p. 125°.

B₂,H₂PtCl₆: leaflets + $4H_2O$ from H_2O. M.p. anhyd. 187° decomp.

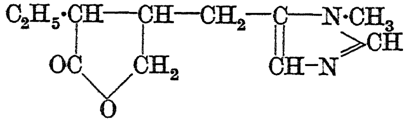
Synthetic product.

Racemic base: colourless cryst. M.p. 128–9°. Sol. $CHCl_3$.

Preobrashenski *et al.*, *Ber.*, 1936, **69**, 1837; 1933, **66**, 1537.

Späth, Kunz, *Ber.*, 1925, **58**, 513.

Pilocarpine



$C_{11}H_{16}O_2N_2$ MW, 208

Alkaloid from leaves of various species of *Pilocarpus*. Colourless oil. B.p. 260°/5 mm. (part. isomerisation). $[\alpha]_D^{20}$ + 100·5° (lowered in presence of alkali). Sol. alkalis ⟶ unstable salts ("pilocarpates"). Heat alone or with alkali ⟶ isopilocarpine.

B,HCl: m.p. 204–5°.

B,HNO₃: *d*-; m.p. 178°; racemic, m.p. 139–40°.

B,HAuCl₄: m.p. anhyd. 130°.

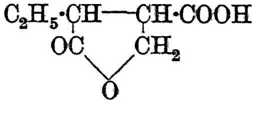
Picrate: m.p. 147°.

Styphnate: m.p. 183°.

Preobrashenski *et al.*, *Ber.*, 1936, **69**, 1835; 1933, **66**, 1187, 1536; 1930, **63**, 460.

Roche Lynch *et al.*, *Analyst*, 1948, **73**, 311.

Pilopic Acid (*Ethylparaconic acid, 2-keto-3-ethyltetrahydrofuran-4-carboxylic acid*)



$C_7H_{10}O_4$ MW, 158

d-.

M.p. 122°. $[\alpha]_D^{18}$ + 54·6° in H_2O.

l-.

M.p. 122°. $[\alpha]_D^{18}$ − 54° in H_2O.

dl-.

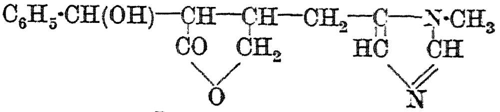
Needles from H_2O. M.p. 90–1° (86–7°). B.p. 210–15°/18 mm., 189–92°/7 mm. slight decomp. Sol. ord. org. solvents.

Et ester: $C_9H_{14}O_4$. MW, 186. Needles from EtOH.Aq. M.p. 49°. B.p. 283°/751 mm. D^{20} 1·1085. Sol. EtOH, Et_2O. Insol. H_2O.

Preobrashenski *et al.*, *Ber.*, 1935, **68**, 847; 1930, **63**, 460.

Welch, *J. Chem. Soc.*, 1931, 1370.

Pilosine



Proposed structure

$C_{16}H_{18}O_3N_2$ MW, 286

Alkaloid from *Pilocarpus microphyllus*. Plates from EtOH. M.p. 187°. Sol. hot H_2O. Very spar. sol. Et_2O, $CHCl_3$, AcOEt, Me_2CO, C_6H_6. $[\alpha]_D$ + 40·2° in $CHCl_3$.

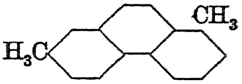
Sulphate: plates from EtOH. M.p. 194–5°. $[\alpha]_D$ + 21·0° in H_2O.

Chloroaurate: golden plates from AcOH. M.p. 143–4°.

Polyakova, Preobrazhenskiĭ, Preobrazhenskiĭ, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 1402.

Pyman, *J. Chem. Soc.*, 1912, 2260.

Pimanthrene (1 : 7-*Dimethylphenanthrene*)



$C_{16}H_{14}$ MW, 206

Plates from EtOH. M.p. 86°.

Picrate: needles from MeOH. M.p. 132°.

Styphnate: needles from MeOH, cryst. from Me_2CO–Et_2O. M.p. 162–4° (159°).

Trinitrobenzene add. comp.: yellow needles from EtOH. M.p. 161·5–162° (159–60°).

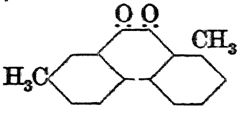
Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1789.

Hosking, McFadyen, *J. Soc. Chem. Ind.*, 1934, **53**, 195T.

Craig, Jacobs, *J. Biol. Chem.*, 1944, **152**, 648.

Jacobs, Huebner, *J. Biol. Chem.*, 1947, **170**, 200.

Pimanthrenequinone (1 : 7-*Dimethylphenanthraquinone*)



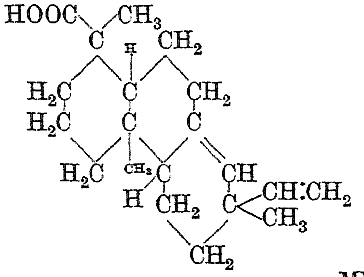
$C_{16}H_{12}O_2$ MW, 236

Plates from EtOH. M.p. 165°.

Quinoxaline deriv.: needles from $CHCl_3$–EtOH. M.p. 194–5°.

Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1789.
Ruzicka, Waldmann, *Helv. Chim. Acta*, 1932, **15**, 913.

d-Pimaric Acid (*Dextropimaric acid*)



$C_{20}H_{30}O_2$ MW, 302

Present in French colophony, etc. Prisms from EtOH. M.p. 218–19° (211°). $[\alpha]_D^{20}$ + 87·3° in $CHCl_3$. Stable to heat and min. acids. Dehydrogenation ⟶ pimanthrene.

Me ester: $C_{21}H_{32}O_2$. MW, 316. B.p. 149–50°/0·03 mm. D_4^{19} 1·030. n_D^{19} 1·52.

Et ester: $C_{22}H_{34}O_2$. MW, 330. B.p. 169–70°/0·2 mm. D_4^{14} 1·013. n_D^{14} 1·5151.

Palkin, Harris, *J. Am. Chem. Soc.*, 1933, **55**, 3677.
Ruzicka, Balas, *Helv. Chim. Acta*, 1923, **6**, 677.
Haworth, *J. Chem. Soc.*, 1932, 2717.
Ruzicka, de Graaff, Goldberg, Frank, *Helv. Chim. Acta*, 1932, **15**, 915.
Harris, Sanderson, *J. Am. Chem. Soc.*, 1948, **70**, 2081.

l-Pimaric Acid.

Note.—Since the compound usually described by this name is not a stereoisomer of *d*-pimaric acid, it has recently been suggested that it should be renamed *l*-sapietic acid, *q.v.*

Pimelic Acid (*Pentane*-1 : 5-*dicarboxylic acid*)

$HOOC{\cdot}CH_2{\cdot}[CH_2]_3{\cdot}CH_2{\cdot}COOH$

$C_7H_{12}O_4$ MW, 160

Prisms from H_2O. M.p. 104–5°. B.p. 212°/10 mm. Sol. H_2O, EtOH, hot C_6H_6. Insol. cold C_6H_6. Non-volatile in steam.

Me ester: $C_8H_{14}O_4$. MW, 174. B.p. 181–2°/18 mm. *Chloride*: $C_8H_{13}O_3Cl$. MW, 192·5. B.p. 135–6°/17 mm.

Di-Me ester: $C_9H_{16}O_4$. MW, 188. B.p. 130–5°/17 mm., 121–2°/11 mm.

Et ester: $C_9H_{16}O_4$. MW, 188. Cryst. from Et_2O. M.p. 10°. B.p. 182°/18 mm., 162°/6 mm. n_D^{20} 1·4415. *Chloride*: $C_9H_{15}O_3Cl$. MW, 206·5. B.p. 139°/17 mm.

Di-Et ester: $C_{11}H_{20}O_4$. MW, 216. B.p. 252–5°/748 mm., 153–6°/24 mm., 139–41°/15 mm. D_4^{20} 0·99448.

Diphenacyl ester: cryst. from EtOH. M.p. 72·4°.

Di-p-*bromophenacyl ester*: cryst. from EtOH. M.p. 136·6°.

Dichloride: $C_7H_{10}O_2Cl_2$. MW, 197. Oil. B.p. 137°/15 mm.

Dinitrile: $C_7H_{10}N_2$. MW, 122. B.p. 175–6°/14 mm. D^{18} 0·949. Solidifies to a glass. Insol. H_2O.

Anilide: cryst. from H_2O. M.p. 108–9°.

Dianilide: cryst. from MeOH.Aq. M.p. 155–6°.

Meyer, *Helv. Chim. Acta*, 1933, **16**, 1292.
Müller, *Monatsh.*, 1934, **65**, 18.
Synder, Brooks, Shapiro, *Organic Syntheses*, Collective Vol. II, 531.
Pummerer, Schönamsgruber, *Ber.*, 1939, **72**, 1834.
Farlow, Whitman, U.S.P. 2,390,576, (*Chem. Abstracts*, 1946, **40**, 1875).
Cass, *Ind. Eng. Chem.*, 1948, **40**, 216.

Pimelic Dialdehyde (*Heptandial*-1 : 7)

$OHC{\cdot}CH_2{\cdot}[CH_2]_3{\cdot}CH_2{\cdot}CHO$

$C_7H_{12}O_2$ MW, 128

Viscous oil with odour resembling tobacco. Very sol. H_2O. Readily polymerises to a white solid insol. H_2O or org. solvents.

Dioxime: cryst. from MeOH. M.p. 153°.

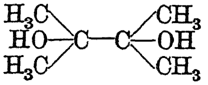
Disemicarbazone: cryst. from EtOH. M.p. 244° decomp.

Weil, Traun, Marcel, *Ber.*, 1922, **55**, 2674.
Fischer, Düll, Ertel, *Ber.*, 1932, **65**, 1472.

Pimelin-ketone.

See Cyclohexanone.

Pinacol (*Pinacone, dimethyl-ψ-butylene glycol, tetramethylethylene glycol*, 2 : 3-*dihydroxy*-2 : 3-*dimethylbutane*)



$C_6H_{14}O_2$ MW, 118

Cryst. from Et_2O, m.p. 38° : cryst. + $6H_2O$ from H_2O, m.p. 47°. B.p. 175°. Sol. EtOH, Et_2O, hot H_2O. Spar. sol. CS_2, cold H_2O.

Monoformyl: b.p. about 90°/20 mm.

Diacetyl: m.p. 65°. Sol. EtOH, Et_2O, $CHCl_3$.

Friedel, Silva, *Ber.*, 1873, **6**, 267.
Allen, Bell, *Organic Syntheses*, 1942, XXII, 40.
Slotterbeck, U.S.P. 2,408,101, (*Chem. Abstracts*, 1947, **41**, 346; *ibid.*, 7277).

Pinacolin (*Pinacolone, methyl* tert.-*butyl ketone*, 1 : 1 : 1-*trimethylacetone*)

$CH_3{\cdot}CO{\cdot}C(CH_3)_3$

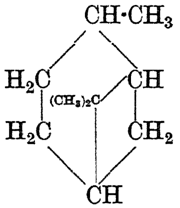
$C_6H_{12}O$ MW, 100

B.p. 106·0–106·1°/760 mm., 105° (103–6°)/746 mm. D^0 0·8265, D_4^{25} 0·8012. n_D^{20} 1·3952–1·3960, n_4^{25} 1·4019.

Oxime: needles from EtOH.Aq. M.p. 78·5–79·5° (74–5°). B.p. 171·6°/748 mm.
Phenylhydrazone: oil. B.p. 165°/32 mm.
2 : 4-*Dinitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 126–7° (125°).
Azine: b.p. 213–16°, 103°/17 mm.

Badertscher, Whitmore, *J. Am. Chem. Soc.*, 1932, **54**, 825.
Allen, Bell, *Organic Syntheses*, 1942, XXII, 39.
Emerson, *J. Am. Chem. Soc.*, 1947, **69**, 1212.
Whitmore, Noll, Meunier, *J. Am. Chem. Soc.*, 1939, **61**, 684.
Amemiya, *J. Chem. Soc. Japan*, 1942, **64**, 1091.

Pinacolin Alcohol.
See Methyl-*tert*.-butylcarbinol.
Pinacolone.
See Pinacolin.
Pinacone.
See Pinacol.
Pinane



$C_{10}H_{18}$ MW, 138

Two stereoisomeric forms are possible but the homogeneity of any of the forms described has not been established.

d-. α-Pinane.
B.p. 166–166·5°/762 mm. D_4^{20} 0·8560. n_D^{20} 1·4630. $[\alpha]_D^{20}$ + 22·83°. Catalytic dehydrogenation ⟶ *o*- and *p*-cymene, ψ-cumene and hemimellitene.

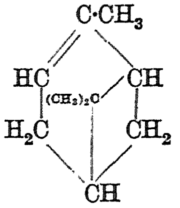
l-. β-Pinane.
B.p. 167·5–168°/748 mm. D_4^{20} 0·8567. n_D^{20} 1·4605. $[\alpha]_D$ − 19·84°.

dl-. *Inactive* Pinane, pinocamphane.
B.p. 164·5–165°. D_4^{20} 0·8551. n_D^{20} 1·4609.

Lipp, *Ber.*, 1923, **56**, 2098.
Vavon, *Compt. rend.*, 1910, **150**, 1127.
Nametkin, Jarzeff, *Ber.*, 1923, **56**, 833.
Rule, Chambers, *J. Chem. Soc.*, 1937, 151.
Komppa, *Chem. Abstracts*, 1947, **41**, 425.
Schmidt, *Chem. Zentr.*, 1942, I, 2531.

3-Pinanol.
See Pinocampheol.
4-Pinanol.
See Verbanol.
3-Pinanone.
See Pinocamphone.
4-Pinanone.
See Verbanone.

α-Pinene



$C_{10}H_{16}$ MW, 136

Constituent of essential oils of Coniferæ. Main constituent of oil of turpentine.

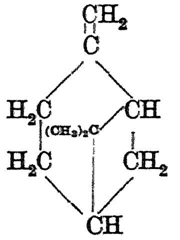
d-. Australene.
M.p. − 50°. B.p. 155–6°, 47–50°/10 mm. D_{15}^{20} 0·862. n_D^{15} 1·4685. $[\alpha]_D$ + 48·3°.
Nitrosochloride: m.p. 81–81·5°. $[\alpha]_D$ + 322°.

l-. Firpene, terebenthene.
B.p. 155–6°. D^{20} 0·8595. n_D^{20} 1·47299. $[\alpha]_D$ − 47·2°.
Nitrosochloride: needles from EtOH–Et_2O. M.p. 81–81·5°. $[\alpha]_D$ − 322° in EtOH or $CHCl_3$.

dl-.
Colourless oil. B.p. 156·2°. D_4^{20} 0·8582. n_D^{20} 1·4658. Heat at 250–70° ⟶ dipentene. Dry HCl ⟶ bornyl chloride.
Nitrosochloride: leaflets from $CHCl_3$–MeOH. M.p. 103°.
Nitrosobromide: cryst. from $CHCl_3$–MeOH. M.p. 91–2° decomp.

Lynn, *J. Am. Chem. Soc.*, 1919, **41**, 361.
Schorger, *J. Am. Chem. Soc.*, 1917, **39**, 1042.
Semmler, Bartelt, *Ber.*, 1907, **40**, 1368.
Wallach, *Ann.*, 1909, **368**, 2; 1890, **258**, 344.
Komppa, *Chem. Abstracts*, 1947, **41**, 435.
Bardyshev *et al.*, *Chem. Abstracts*, 1948, **42**, 9201.
Hückel, *Chem. Abstracts*, 1943, **37**, 3074.

β-Pinene (*Nopinene, pseudopinene*)



$C_{10}H_{16}$ MW, 136

Constituent of oil of turpentine. B.p. 163–4°. D_4^{20} 0·8694, D^{22} 0·8675. n_D^{20} 1·4782, n_D^{20} 1·4749. $[\alpha]_D$ −22° (− 18·2°). Dry HCl ⟶ bornyl chloride + dipentene dihydrochloride.

Vavon, *Compt. rend.*, 1910, **150**, 1129.
Bardyshev *et al.*, *Chem. Abstracts*, 1948, **42**, 9201.
Bonnet, *Chem. Abstracts*, 1939, **33**, 4223.
Komppa, *Chem. Abstracts*, 1947, **41**, 425.

Pinene Hydrate.
See Methylnopinol.

"Pinene hydrochloride."
See Bornyl chloride.

Pinic Acid (2 : 2-*Dimethyl-3-carboxycyclobutylacetic acid,* 2 : 2-*dimethyl-3-carboxymethylcyclobutane-1-carboxylic acid*)

CH_2—$CH{\cdot}CH_2{\cdot}COOH$
HOOC·CH——$C(CH_3)_2$

$C_9H_{14}O_4$ MW, 186

Trans :

d-.
Prisms from Et_2O–pet. ether. M.p. 135–6°. B.p. 212–16°/10 mm. Very sol. H_2O, Et_2O. Sol. Me_2CO. Spar. sol. $CHCl_3$, C_6H_6. Insol. pet. ether. $[\alpha]_D$ + 7·1° in Me_2CO.
Di-Me ester : $C_{11}H_{18}O_4$. MW, 214. Liq. B.p. 128–30°/9 mm. D^{20} 1·0548. n_D^{20} 1·4487. $[\alpha]_D$ + 13·8°.
Di-Et ester : $C_{13}H_{22}O_4$. MW, 242. Liq. B.p. 142–6°/10 mm. D^{20} 1·0104. n_D 1·44962. $[\alpha]_D$ + 8·0°.

l-.
Needles from H_2O. M.p. 135–6°. $[\alpha]_D$ − 7·1° in Me_2CO.

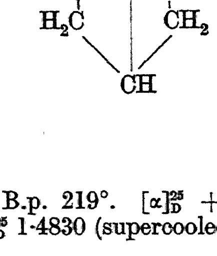
dl-.
Prisms from H_2O. M.p. 101–102·5°. B.p. 214–16°/9 mm. Spar. sol. cold H_2O.
Di-Me ester : liq. B.p. 134–8°/17·5 mm. D^{20} 1·053. n_D^{20} 1·4490.
Di-Et ester : liq. B.p. 145–7°/10 mm. D^{20} 1·0093. n_D 1·44662.
Diamide : m.p. 222–3°.
Dianilide : m.p. 204°.

Semmler, Mayer, *Ber.*, 1911, **44**, 3665.
Barbier, Grignard, *Bull. soc. chim.*, 1910, **7**, 548.
Baeyer, *Ber.*, 1896, **29**, 326.
Guha, Ganapathi, Subramanian, *Ber.*, 1937, **70**, 1505.
Grandperrin, *Ann. chim.*, 1936, **6**, 5.

Pinite.
See under Inositol.

Pinitol.
See under Inositol.

Pinocampheol (3-*Pinanol*)



$C_{10}H_{18}O$ MW, 154

Cis :

d-.
M.p. 57°. B.p. 219°. $[\alpha]_D^{25}$ + 39° in Et_2O. D^{15} 0·973. n_D^{25} 1·4830 (supercooled).
Acetyl : b.p. 227–8°/760 mm., 80–2°/2–3 mm. D_4^{25} 0·979. n_D^{21} 1·4641. $[\alpha]_D^{25}$ + 23°.
Phthalate : m.p. 126°.
1-*Naphthylurethane* : cryst. from pet. ether. M.p. 87·5–88° corr.

l-.
M.p. 57°. B.p. 219°. $[\alpha]_D$ −36°. D^{15} 0·973.
Acetyl : b.p. 82–4°/3 mm. D_4^{20} 0·9781. n_D^{20} 1·4638.
Phthalate : m.p. 126°.
Naphthylurethane : m.p. 88°.

dl-.
M.p. 42°. B.p. 219°. D^{15} 0·973.

Trans :

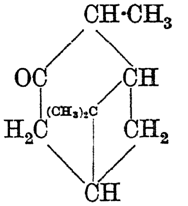
d-.
M.p. 67°. B.p. 217°. D^{15} 0·968. n_D^{20} 1·48330. $[\alpha]_D$ + 55°.
Phenylurethane : m.p. 77°.

l-.
M.p. 67°. B.p. 217°. D^{15} 0·968. n_D^{20} 1·48335. $[\alpha]_D$ − 55°.
Phthalate : m.p. 107°.
Phenylurethane : m.p. 77°.
Naphthylurethane : m.p. 91°.

dl-.
M.p. 36°. B.p. 217°. D^{15} 0·968.
Phthalate : m.p. 113°.
Phenylurethane : m.p. 99°.

Schmidt, Schulz, *Chem. Zentr.*, 1934, II, 2077.
Stallcup, Hawkins, *J. Am. Chem. Soc.*, 1942, **64**, 1808.
Schmidt, *Ber.*, 1944, **77**, 544.
Treibs, *ibid.*, 572.

Pinocamphone (3-*Pinanone*)



$C_{10}H_{16}O$ MW, 152

Cis :

d-.
B.p. 213·5–214°, 70°/5 mm. D^{15} 0·9688. n_D 1·47495. $[\alpha]_D$ + 11°.
Semicarbazone : m.p. 219–20°.

Trans :

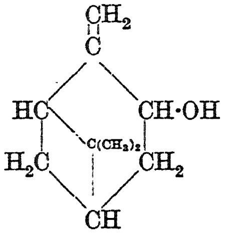
d-.
B.p. 211–211·5°, 70–2°/5 mm. D^{15} 0·9643. n_D^{20} 1·47279. $[\alpha]_D$ + 24°.
Semicarbazone : m.p. 227°.
Oxime : m.p. 56°.

l-.
B.p. 212°. D_4^{25} 0·964. n_D^{25} 1·4735. $[\alpha]_D^{25}$ − 13·5°.

Semicarbazone : needles from MeOH.Aq. M.p. 227·5–228°.

Stallcup, Hawkins, *J. Am. Chem. Soc.*, 1942, **64**, 1807.
Schmidt, *Chem. Zentr.*, 1942, I, 2531; *Chem. Abstracts*, 1943, **37**, 4380.
Schmidt, Schulz, *Chem. Abstracts*, 1935, **29**, 6225.

Pinocarveol (*Pinyl alcohol, isocarveol*)



$C_{10}H_{16}O$ MW, 152

Cis :

d-.

M.p. 51°. B.p. 217–18°/760 mm. D^{20} 1·004. n_D^{20} 1·50568. $[\alpha]_D$ + 55° in 5% EtOH. Dehydrates on shaking with 10% H_2SO_4. Does not form a solid dibromide, phthalate, or phenylurethane.

Acetyl: b.p. 231–2°/760 mm. D^{15} 1·003. n_D^{20} 1·48023. $[\alpha]_D$ + 64°.

p-*Nitrobenzoyl* : m.p. 104°. $[\alpha]_D$ + 136°.

Trans :

l-.

Occurs in oil from *Eucalyptus globulus*. M.p. about 5°. B.p. 209–10°/760 mm. D^{20} 0·981. n_D^{20} 1·50054. $[\alpha]_D$ − 72°. Becomes turbid in air. Forms a hydrate on shaking with 10% H_2SO_4.

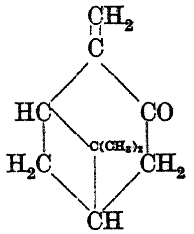
Hydrate : m.p. 196°.

Acetyl : b.p. 227–8°. D^{15} 0·999. $[\alpha]_D$ + 16·3°.

1-*Naphthylurethane* : m.p. 95°.

Schmidt, *Ber.*, 1944, **77**, 167.

Pinocarvone (*Carvopinone*)



$C_{10}H_{14}O$ MW, 150

d-.

Oil. Prepared by oxidising *d*-pinocarveol. B.p. 84–5°/4 mm. D_4^{25} 0·981. n_D^{20} 1·4935. $[\alpha]_D$ + 63°.

Oxime : m.p. 132–3°.

l-.

Occurs in Spanish eucalyptus oil (*E. globulus*). Oil. F.p. − 1·8°. B.p. 67–9°/4 mm. D^{15} 0·9875. n_D^{20} 1·49498. $[\alpha]_D$ − 68·5°.

Semicarbazone : m.p. 350°.

Oxime : two forms. (i) Liquid. $[\alpha]_D$ − 11°. (ii) Solid. M.p. 131–2°.

Dibromide : m.p. 73·5–74°. $[\alpha]_D$ − 130°.

dl-.

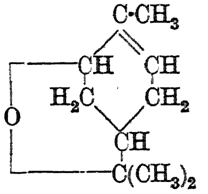
The so-called carvopinone of Wallach (*Ann.*, 1906, **346**, 231) is *dl*-pinocarvone, b.p. 94–6°/12 mm.

Oxime : m.p. 131–2°. This oxime is the nitrosopinene of Wallach (*loc. cit.*).

Note. The so-called pinocarvone obtained by Wallach (*Ann.*, 1906, **346**, 220) from essential oil is myrtenal, *q.v.*

Schmidt, *Chem. Zentr.*, 1942, I, 2530; *Ber.*, 1944, **77**, 167.

Pinol



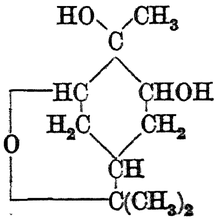
$C_{10}H_{16}O$ MW, 152

Liq. with odour resembling cineol. B.p. 183–4°, 76–7°/14 mm. D^{20} 0·953. n_D^{20} 1·4695. $KMnO_4 \longrightarrow$ terpenylic acid.

Nitrosochloride : m.p. 103°.

Neave, *J. Chem. Soc.*, 1912, **101**, 514.
Wallach, *Ann.*, 1896, **291**, 349.
Wagner, *Ber.*, 1894, **27**, 1644.

Pinol Glycol



$C_{10}H_{18}O_3$ MW, 186

Cis :

Needles from $CHCl_3$. M.p. 125°. B.p. 157°/12 mm.

Di-Et ether : $C_{14}H_{26}O_3$. MW, 242. Needles from Et_2O. M.p. 52–3°.

Diacetyl : needles from H_2O. M.p. 97–8°.

Trans :

Two forms :

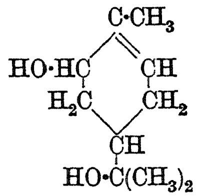
(i) *Active*. M.p. 74°.

(ii) *Inactive*. Plates from H_2O. M.p. 128–9°. B.p. 157–8°/12 mm.

Diacetyl : m.p. 37–8°. B.p. 165–7°/17 mm.

Wallach, *Ann.*, 1892, **268**, 223; 1890, **259**, 311.
Wagner, Slawinski, *Ber.*, 1899, **32**, 2066; 1894, **27**, 1644.

Pinol Hydrate (dl-Δ^1-p-*Menthenediol*-6 : 8)



$C_{10}H_{18}O_2$ MW, 170

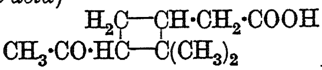
Plates or needles. M.p. 131°. B.p. 270–1°. Sol. H_2O, EtOH, Et_2O. $H_2SO_4 \longrightarrow$ red col.

Diacetyl: b.p. 159–161·5°. D_0^{18} 1·0385.

Wallach, *Ann.*, 1896, **291**, 351.
Henderson, Agnew, *J. Chem. Soc.*, 1909, **95**, 291.

Pinonene.
See *d*-Δ^4-Carene.

Pinonic Acid (2 : 2-*Dimethyl*-3-*acetocyclobutylacetic acid*)



$C_{10}H_{16}O_3$ MW, 184

d-.
Cryst. from pet. ether. M.p. 69°. B.p. 168°/12 mm. $[\alpha]_D^{25}$ + 92·6° in $CHCl_3$.
Oxime: two forms. (i) Plates from MeOH. M.p. 129°. (ii) Cryst. from AcOH. M.p. 190–1°.
Semicarbazone: cryst. from EtOH. M.p. 204°.

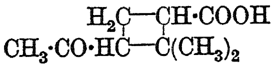
l-.
Prisms from H_2O or C_6H_6–pet. ether. M.p. 68–9°. $[\alpha]_D^{22}$ − 90·6° in $CHCl_3$.
Oxime: two forms. (i) Plates from Et_2O–pet. ether. M.p. 129°. (ii) Cryst. from AcOH.Aq. M.p. 204°.

dl-.
Plates or prisms from MeOH. M.p. 103–5°. B.p. 180–7°/14 mm. Sol. $CHCl_3$.
Oxime: prisms from MeOH. M.p. 150°.
Semicarbazone: m.p. 206–7°.
Me ester: $C_{11}H_{18}O_3$. MW, 198. B.p. 254–5°/755 mm., 128°/11 mm. n_D^{17} 1·4558.

Note.—Numerous oily forms are known which are mixtures of stereoisomers. *Cis*- pinonic acid gives *trans*-pinic acid on hypobromite oxidation.

Schmidt, *Z. angew. Chem.*, 1929, **42**, 126.
Ruzicka, Trebler, *Helv. Chim. Acta*, 1920, **3**, 762.
Barbier, Grignard, *Bull. soc. chim.*, 1910, **7**, 553.
Perkin, Simonsen, *J. Chem. Soc.*, 1909, **95**, 1175.
Baeyer, *Ber.*, 1896, **29**, 23, 2786.
Komppa, Klami, Kuvaja, *Ann.*, 1941, **547**, 185.
Komppa, *Chem. Abstracts*, 1947, **41**, 425.
Briggs, Taylor, *J. Org. Chem.*, 1947, **12**, 555.

Pinononic Acid (2 : 2-*Dimethyl*-3-*acetocyclobutane*-1-*carboxylic acid*)



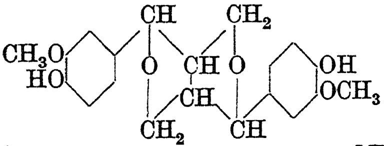
$C_9H_{14}O_3$ MW, 170

d-.
Prisms from H_2O. M.p. 129°. $[\alpha]_D$ + 40·2° in Et_2O.
Semicarbazone: m.p. 212° (204°).

l-.
Prisms from $CHCl_3$. M.p. 129°.
Oxime: prisms from Et_2O. M.p. 178–80°.
Me ester: $C_{10}H_{16}O_3$. MW, 184. B.p. 127°/13 mm.

Guha, Ganapathi, *Chem. Abstracts*, 1935, **29**, 5818.
Blumann, Zeitschel, *Ber.*, 1913, **46**, 1189.
Kerschbaum, *Ber.*, 1900, **33**, 891.
Wagner, Ertschikowsky, *Ber.*, 1896, **29**, 881.

***d*-Pinoresinol**



$C_{20}H_{22}O_6$ MW, 358

Constituent of fir resin. Cryst. from EtOH.Aq. M.p. 122°. $[\alpha]_D^{21}$ + 84·4° in Me_2CO.
Diacetyl: m.p. 166–167·5°. $[\alpha]_D^{20}$ + 50·9° in Me_2CO, + 49·1° in $CHCl_3$.
Dibenzoyl: m.p. 163–4°. $[\alpha]_D^{20}$ + 42·8° in Me_2CO, + 46·9° in $CHCl_3$.
Di-Me ether: $C_{22}H_{26}O_6$. MW, 386. M.p. 107–8°. $[\alpha]_D^{20}$ + 64·5° in $CHCl_3$.
Di-Et ether: $C_{24}H_{30}O_6$. MW, 414. M.p. 122°. $[\alpha]_D^{20}$ + 62° in $CHCl_3$.

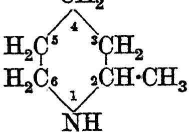
Bamberger, Landsiedl, *Monatsh.*, 1897, **18**, 481.
Kaku, Ri, *J. Pharm. Soc. Japan*, 1937, **57**, 1015.
Erdtmann, *Ann.*, 1935, **516**, 162.
Haworth, *J. Chem. Soc.*, 1942, 448.

Pinosylvin.
See 3 : 5-Dihydroxystilbene.

α-Pipecolein.
See 6-Methyl-1 : 2 : 3 : 4-tetrahydropyridine.

Pipecolic Acid.
See Hexahydropicolinic Acid.

α-Pipecoline (2-*Methylpiperidine, hexahydro-α-picoline*)



$C_6H_{13}N$ MW, 99

d-.
B.p. 117–117·5°. $[\alpha]_D^{15}$ + 18·7° in $CHCl_3$. n_D^{15} 1·44983.
B,HCl: m.p. 210°.
B,HAuCl₄: m.p. 131–2°.
B_2,H_2PtCl_6: m.p. 194°.
Picrate: m.p. 116–17°.
d-*Tartrate*: m.p. anhyd. 110–12°.
l-*Tartrate*: m.p. anhyd. 126°.
N-*Me*: $C_7H_{15}N$. MW, 113. B.p. 127°. D^{16} 0·825. $[\alpha]_D^{15}$ + 68·8°. *Picrate*: cryst. from MeOH. M.p. 240° decomp.

l-.
B,HCl: m.p. 190°.
$B,HAuCl_4$: m.p. 131–2°.
B_2,H_2PtCl_6: m.p. 194°.
Picrate: m.p. 116–17°.
d-*Tartrate*: m.p. anhyd. 126°.
l-*Tartrate*: m.p. anhyd. 111–12°.
N-*Me*: $[\alpha]_D$ − 33·6°.

dl-.
Liq. resembling piperidine in odour. B.p. 117–18°/747 mm. $D_4^{23\cdot6}$ 0·8436. $n_D^{23\cdot6}$ 1·4464. Sol. H_2O, EtOH, Et_2O.
B,HCl: prisms from H_2O. M.p. 210°.
B,HBr: needles. M.p. 189°.
$B,HAuCl_4$: yellow prisms. M.p. 127° (118–19°).
B_2,H_2PtCl_6: m.p. 202°.
Picrate: yellow needles. M.p. 134–5°.
N-p-*Toluenesulphonyl*: cryst. from ligroin. M.p. 54·5–55°.
N-*Benzoyl*: cryst. from EtOH. M.p. 44–5°.
N-*Me*: b.p. 126–7°/742 mm. D_4^{15} 0·824. n_D^{20} 1·4395. Sol. EtOH, Et_2O, CS_2. Sol. 10–12 parts cold H_2O. *Picrate*: cryst. from EtOH. M.p. 233°. *B,HCl*: prisms from EtOH. M.p. 258–9°. $B,HAuCl_4$: m.p. 215–16°. B_2,H_2PtCl_6: orange red prisms from MeOH. M.p. 178–181·5°.
N-*Et*: $C_8H_{17}N$. MW, 127. B.p. 147–8°. $D^{17\cdot5}$ 0·8368. $n_D^{24\cdot5}$ 1·4480. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O. $B,HAuCl_4$: plates from EtOH.Aq. M.p. 108°.
N-*Propyl*: $C_9H_{19}N$. MW, 141. B.p. 167–167·5°. D^{20} 0·8296. *Picrate*: m.p. 113°.
N-*Amyl*: b.p. 92–3°/16 mm. n_D^{25} 1·4500. *Picrate*: m.p. 94–5°. *B,HCl*: m.p. 166·5–167·5°.
N-*Phenyl*: b.p. 133°/14 mm. D_{15}^{17} 0·987. n_D^{13} 1·5543. *Picrate*: m.p. 157–8°.
N-*Benzyl*: b.p. 267°, 160–2°/47 mm.
N-*Nitroso*: b.p. 123°/31 mm.

Borissow, *Ber.*, 1930, **63**, 2278.
Adkins, Kuick, Farlow, Wojcik, *J. Am. Chem. Soc.*, 1934, **56**, 2425.
Skita, Brunner, *Ber.*, 1916, **49**, 1601.
Ladenburg, *Ber.*, 1898, **31**, 291.
Lipp, *Ann.*, 1896, **289**, 225.
Leithe, *Ber.*, 1930, **63**, 805.

β-Pipecoline (3-*Methylpiperidine, hexahydro-β-picoline*).

d-.
d-*Tartrate*: m.p. 76–8°.
l-*Tartrate*: m.p. 170°.
N-*Me*: *picrate*: cryst. from EtOH. M.p. 176–8°. $[\alpha]_{5780}^{23}$ + 97°. *Methiodide*: m.p. 199–201°.

l-.
B.p. 124°. $[\alpha]_D^{25}$ − 4°.
d-*Tartrate*: needles. M.p. 170–2°.
N-*Me*: *picrate*: needles from EtOH. M.p. 176–9°. $[\alpha]_{5780}^{23}$ − 12·6°. *Methiodide*: needles from EtOH. M.p. 200–201°. $[\alpha]_D^{28}$ + 7·0°.
N-3 : 5-*Dinitrobenzoyl*: needles from EtOH. M.p. 114–16°. $[\alpha]_D^{32}$ − 30°.

dl-.
Liq. resembling piperidine in odour. B.p. 125–6°/763 mm. $D_4^{24\cdot3}$ 0·8446. $n_D^{24\cdot3}$ 1·4463. Very sol. H_2O.
B,HCl: needles from C_6H_6. M.p. 171–2°.
B,HI: needles from C_6H_6. M.p. 158–9°.
$B,HAuCl_4$: m.p. 130–1°.
B_2,H_2PtCl_6: red prisms from H_2O. M.p. 207°.
Picrate: yellow prisms. M.p. 136–8°.
d-*Tartrate*: m.p. 144–6°.
N-*Me*: oil. B.p. 124–6°. D^{15} 0·818. *Picrate*: cryst. from EtOH. M.p. 165–8°. B_2,H_2PtCl_6: orange prisms. M.p. 156–8°. *Methiodide*: cryst. from EtOH. M.p. 196–7°.
N-*Et*: b.p. 145·5–146·5°.
N-p-*Nitrophenyl*: leaflets from EtOH. M.p. 61°.
N-2 : 4-*Dinitrophenyl*: yellow needles. M.p. 67°.

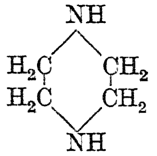
Franke, Kohn, *Monatsh.*, 1879, **23**, 878, 883.
Ladenburg, Hesekiel, *Ann.*, 1888, **247**, 67.
Ladenburg, *Ber.*, 1894, **27**, 75.

γ-Pipecoline (4-*Methylpiperidine, hexahydro-γ-picoline*).
Liq. which fumes in air. B.p. 126·5–129°. D^0 0·8674. Sol. H_2O.
B,HCl: m.p. 186–189·5°.
Chloroaurate: yellow needles from H_2O. M.p. 125–7°.
N-*Amyl*: b.p. 83–4°/10 mm. n_D^{25} 1·4443. *B,HCl*: m.p. 239–41°.
N-*Benzyl*: b.p. 128–9°/14 mm. n_D^{25} 1·5126. *B,HCl*: m.p. 166·5–168°.
N-m-*Nitrobenzoyl*: m.p. 72–3°.

Ladenburg, *Ann.*, 1888, **247**, 69.
Paden, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 2490.

Pipecolinic Acid.
See Hexahydropicolinic Acid.

Piperazine (*Diethylenediamine, hexahydropyrazine*)



$C_4H_{10}N_2$ MW, 86

Hygroscopic plates from EtOH. M.p. 104°. B.p. 140°.

Hydrate: cryst. + $6H_2O$. M.p. 44°. B.p. 125–30°. Sol. H_2O, EtOH. Insol. Et_2O. $k = 6{\cdot}4 \times 10^{-5}$ at 25°. Aq. sol. reacts strongly alkaline. Dist. with Zn ⟶ pyrazine.

B,2C₆H₅OH: prisms from EtOH. M.p. 99–101°.

Picrate: yellow needles from H_2O. M.p. 280° decomp.

N-*Me*: $C_5H_{12}N_2$. MW, 100. B.p. 134–6°. *B,2HCl,H₂O*: cryst. M.p. 82·5–83°. *Dipicrate*: m.p. 272° decomp.

N : N-*Di-Me*: $C_6H_{14}N_2$. MW, 114. B.p. 131–2°. D_4^{20} 0·8600. n_α^{20} 1·4474. *B,2HCl*: cryst. with $\frac{2}{3}H_2O$. M.p. 251·5–253°. *Picrate*: m.p. 280°.

N-*Et*: *B,2HCl*: cryst. from EtOH. M.p. 203–5°.

N : N-*Di-Et*: $C_8H_{18}N_2$. MW, 142. B.p. 169–71° (165°). *B,2HCl*: needles from EtOH. M.p. 277° decomp.

N : N-*Dipropyl*: $C_{10}H_{22}N_2$. MW, 170. B.p. 206°. *Dipicrate*: m.p. 258° decomp.

N-*Amyl*: b.p. 105–10°/9 mm. n_D^{20} 1·4650–9. *B,2HCl*: rods from isopropyl alcohol. M.p. 267–9°.

N-*Phenyl*: *see* *N*-Phenylpiperazine.

N : N-*Diphenyl*: *see* 1 : 4-Diphenylpiperazine.

N : N-*Di-o-tolyl*: $C_{18}H_{22}N_2$. MW, 266. Needles from EtOH. M.p. 174°.

N : N-*Di-m-tolyl*: plates from EtOH. M.p. 126°.

N : N-*Di-p-tolyl*: prisms from pet. ether. M.p. 189–90°.

N-*Benzyl*: b.p. 128–35°/6 mm. *B,2HCl*: flat needles from EtOH. M.p. 253°.

N : N-*Dibenzyl*: $C_{18}H_{22}N_2$. MW, 266. Needles from EtOH. M.p. 92°.

N-*Acetyl*: m.p. 52°. *B,HCl*: m.p. 181°.

N : N-*Diacetyl*: hygroscopic needles or plates from pet. ether. M.p. 144° (138°).

N-*Benzoyl*: m.p. 75°. *B,HCl*: m.p. 274°.

N : N-*Dibenzoyl*: m.p. 196°.

N : N-*Dibenzenesulphonyl*: cryst. from $PhNO_2$. M.p. 291·3–291·7°.

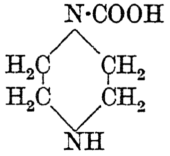
N-p-*Toluenesulphonyl*: m.p. 173° after sintering at 168°.

N : N-*Di*-p-*Toluenesulphonyl*: cryst. from $PhNO_2$. M.p. 298·4–298·6°.

N : N-*Dinitroso*: yellow plates from H_2O. M.p. 158°.

Garelli, Racciu, *Angew. Chem.*, 1934, **47**, 366.
Abderhalden, Klarmann, Schwab, *Z. physiol. Chem.*, 1924, **135**, 180.
Pratt, Young, *J. Am. Chem. Soc.*, 1918, **40**, 1429.
Prelog, Stepán, *Chem. Abstracts*, 1935, **29**, 4013.
Jacobi, *Ber.*, 1933, **66**, 113.
Abderhalden, Haas, *Z. physiol. Chem.*, 1925, **148**, 245.
Kitchen, Pollard, *J. Am. Chem. Soc.*, 1947, **69**, 854.
Martin, Martell, *J. Am. Chem. Soc.*, 1948, **70**, 1817.

Piperazine-*N*-carboxylic Acid



$C_5H_{10}O_2N_2$ MW, 130

Cryst. M.p. 162–5° (sealed tube).

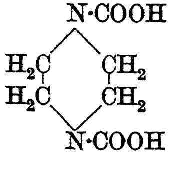
Me ester: $C_6H_{12}O_2N_2$. MW, 144. B.p. 112–16°/7 mm. N-*Me*: m.p. 116–21°.

Et ester: $C_7H_{14}O_2N_2$. MW, 158. B.p. 237°, 116–17°/12 mm. *Hydrochloride*: m.p. 145°. N'-*Me*: b.p. 97–8°/8 mm. N'-*Et*: b.p. 132°/28 mm. N'-*Propyl*: b.p. 136°/16 mm. N'-*Benzoyl*: cryst. from ligroin. M.p. 82°. N'-p-*Toluenesulphonyl*: cryst. from C_6H_6-ligroin. M.p. 121°.

Butyl ester: b.p. 141–3°/10 mm.

Moore, Boyle, Thorn, *J. Chem. Soc.*, 1929, 39.
Rosdalsky, *J. prakt. Chem.*, 1896, **53**, 24.

Piperazine-*N* : *N'*-dicarboxylic Acid



$C_6H_{10}O_4N_2$ MW, 174

Free acid unknown.

Di-Me ester: $C_8H_{14}O_4N_2$. MW, 202. Cryst. from H_2O. M.p. 81°. B.p. 163°/11 mm.

Di-Et ester: $C_{10}H_{18}O_4N_2$. MW, 230. Cryst. from hexane. M.p. 49°. B.p. 131–33°/3 mm.

Dibutyl ester: b.p. 205–8°/10 mm.

Diphenyl ester: $C_{18}H_{18}O_4N_2$. MW, 326. Prisms. M.p. 177–8°.

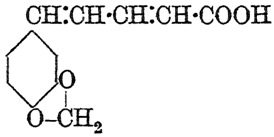
Di-1-naphthyl ester: $C_{26}H_{22}O_4N_2$. MW, 426. M.p. 190–1°.

Di-2-naphthyl ester: m.p. 220°.

Dinitrile: $C_6H_8N_4$. MW, 136. Cryst. from EtOH. M.p. 168°.

van Dorp, *Rec. trav. chim.*, 1909, **28**, 75.
Cazeneuve, Moreau, *Compt. rend.*, 1897, **125**, 1183.

Piperic Acid (4-[3 : 4-*Methylenedioxyphenyl*]-1 : 4-*butadiene*-1-*carboxylic acid*, 3-*piperonylidenecrotonic acid*, 4-[3 : 4-*methylenedioxyphenyl*]-*vinylacrylic acid*)



$C_{12}H_{10}O_4$ MW, 218

Colourless needles from EtOH, turning yellow in light. M.p. 215°. Sol. hot EtOH. Spar. sol. Et_2O, C_6H_6. Insol. H_2O. Sublimes in yellow needles. $H_2SO_4 \longrightarrow$ bluish-violet col. $KMnO_4$ at 3° $\longrightarrow$ piperonal.

Me ester: $C_{13}H_{12}O_4$. MW, 232. Yellow plates from MeOH. M.p. 146°.

Et ester: $C_{14}H_{14}O_4$. MW, 246. Plates from EtOH. M.p. 78°.

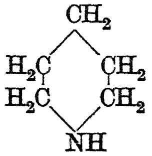
p-*Nitrobenzyl ester*: cryst. from 80% EtOH. M.p. 145°.

Ladenburg, Scholtz, *Ber.*, 1894, **27**, 2959.
Babo, Keller, *J. prakt. Chem.*, 1857, **72**, 56.
Fittig, Mielch, *Ann.*, 1869, **152**, 27.

Piperideine.

See Tetrahydropyridine.

Piperidine (*Hexahydropyridine, pentamethyleneimine*)



$C_5H_{11}N$ MW, 85

Occurs in form of piperine in black pepper (*Piper nigrum, P. longum, P. officinarum*). Colourless liq. with characteristic odour. M.p. − 9°. B.p. 106·0°, 52·6°/170 mm., 17·7°/20 mm. D_4^{20} 0·8606, D_4^{50} 0·8336, D_4^{80} 0·8033. n_D^{20} 1·4530. Heat of comb. C_v 825 Cal. $k = 1{\cdot}58 \times 10^{-3}$ at 25°. Misc. with H_2O in all proportions. Pyrolysis $\longrightarrow$ pyrrole. Forms a hydrate, m.p. − 14°. Forms complexes with heavy metal salts.

B,HCl: prisms from EtOH. M.p. 244–5°.

B,HBr: prisms from $CHCl_3$. M.p. 235°.

B,HNO₂: plates. M.p. 110°.

Acetate: m.p. 106°.

Picrate: yellow needles from H_2O. M.p. 151-2°.

Styphnate: m.p. 231–2°.

B,HAuCl₄: plates from EtOH. M.p. 204°.

N-*Me*: *see* N-Methylpiperidine.

N-*Et*: *see* N-Ethylpiperidine.

N-*Propyl*: $C_8H_{17}N$. MW, 127. B.p. 149–50°. D^{20} 0·8231. n_D^{20} 1·4446. *B,HCl*: m.p. 225°. *Picrate*: m.p. 108°. *Methiodide*: m.p. 181–2°. *Ethiodide*: m.p. 276·5°.

N-*Isopropyl*: b.p. 149–50°. *Picrate*: m.p. 153°.

N-*Butyl*: $C_9H_{19}N$. MW, 141. B.p. 175–6°. D^{20} 0·8245. n_D^{20} 1·4467. *B,HCl*: m.p. 240°. *Picrate*: m.p. 133°. *Methiodide*: m.p. 198°.

N-*Amyl*: $C_{10}H_{21}N$. MW, 155. B.p. 196°. D^{20} 0·8282. n_D^{20} 1·4498. *B,HCl*: m.p. 217°. *Picrate*: m.p. 107°.

N-*Isoamyl*: b.p. 188°. *Methiodide*: m.p. 195°.

N-*Allyl*: $C_8H_{15}N$. MW, 125. B.p. 151–2°. D^{19} 0·8445.

N-*Benzyl*: $C_{12}H_{17}N$. MW, 175. B.p. 245°, 119°/13 mm. B_2,H_2PtCl_6: m.p. 191–3°. *Picrate*: m.p. 166°. *Picrolonate*: m.p. 240°.

N-*Phenyl*: *see* N-Phenylpiperidine.

N-p-*Tolyl*: *picrate*: cryst. from EtOH. M.p. 176°.

N-*Picryl*: yellow prisms from EtOH. M.p. 104–6°.

N-*Triphenylmethyl*: needles from EtOH. M.p. 153°.

N-*Formyl*: piperidine-N-aldehyde. $C_6H_{11}ON$. MW, 113. B.p. 222°. *B,HBr*: m.p. 103·5°.

N-*Acetyl*: b.p. 226–7°. B,H_2PtCl_6: red cryst. M.p. 107–9°.

N-*Benzoyl*: m.p. 48°. B.p. 320–1°, 180–4°/20 mm.

N-p-*Bromobenzoyl*: plates from EtOH. M.p. 95°.

N-o-*Nitrobenzoyl*: yellow cryst. from EtOH. M.p. 38–9°. *B,HCl*: cryst. from EtOH. M.p. 210·5–212°.

N-m-*Nitrobenzoyl*: yellow oil. *B,HCl*: cryst. from EtOH. M.p. 202·5–205°.

N-p-*Nitrobenzoyl*: yellow cryst. from EtOH. M.p. 120·5°.

N-3 : 5-*Dinitrobenzoyl*: cryst. from EtOH. M.p. 147°.

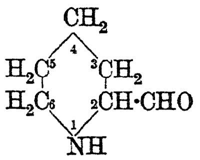
N-*Benzenesulphonyl*: prisms. M.p. 93–4°.

N-p-*Toluenesulphonyl*: plates from dioxan. M.p. 130°.

N-*Nitroso*: yellow oil. B.p. 218°, 109°/20 mm.

Marvel, Lazier, *Organic Syntheses*, Collective Vol. I, 93.
Skita, Brunner, *Ber.*, 1916, **49**, 1600.
Babo, Keller, *J. prakt. Chem.*, 1857, **72**, 55.
Marie, Lejeune, *J. chim. phys.*, 1925, **22**, 59.
Skita, Meyer, *Ber.*, 1912, **45**, 3592.
Wojcik, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 2419.
Staudinger, Müller, *Ber.*, 1923, **56**, 711.
Ouchakof, Lifchitz, Jdanova, *Bull. soc. chim.*, 1935, **2**, 573.

Piperidine-2-aldehyde (*2-Formylpiperidine, 2-aldehydopiperidine*)



$C_6H_{11}ON$ MW, 113

Di-Et acetal: $C_{10}H_{21}O_2N$. MW, 187. B.p. 95–105°/14 mm.

p-*Nitrophenylhydrazone hydrochloride*: yellow cryst. M.p. 228° decomp.

Harries, Lénart, *Ann.*, 1915, **410**, 105.

Piperidine-3-aldehyde (*3-Formylpiperidine, 3-aldehydopiperidine*).

N-*Et*: $C_8H_{15}ON$. MW, 141. B.p. 40°/0·2 mm. *Di-Et acetal*: $C_{12}H_{25}O_2N$. MW, 215. B.p. 63–5°/0·04 mm.

Di-Et acetal: b.p. 104°/9 mm.

m-*Nitrophenylhydrazone hydrochloride*: yellow cryst. M.p. 232–3° decomp.

Wohl, Losanitsch, *Ber.*, 1907, **40**, 4695.

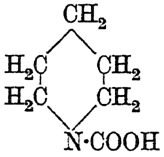
Piperidine-*N*-aldehyde.

See under Piperidine.

Piperidine-carboxylic Acid.

See Hexahydroisonicotinic Acid, Hexahydronicotinic Acid *and* Hexahydropicolinic Acid.

Piperidine-*N*-carboxylic Acid



$C_6H_{11}O_2N$ MW, 129

Free acid unknown.

Me ester: $C_7H_{13}O_2N$. MW, 143. B.p. 201°.

Et ester: pentamethyleneurethane. $C_8H_{15}O_2N$. MW, 157. B.p. 211–12°, 103°/20 mm.

Phenyl ester: $C_{12}H_{15}O_2N$. MW, 205. Plates from EtOH. M.p. 80°.

2-*Naphthyl ester*: $C_{16}H_{17}O_2N$. MW, 255. Needles from EtOH. M.p. 107°.

Chloride: $C_6H_{10}ONCl$. MW, 147·5. B.p. 237–8°, 112°/13 mm.

Amide: pentamethyleneurea. $C_6H_{12}ON_2$. MW, 128. Needles from EtOH. M.p. 105–6°.

Nitrile: *N*-cyanopiperidine. $C_6H_{10}N_2$. MW, 110. B.p. 124°/30 mm., 102°/10 mm.

Schotten, *Ber.*, 1883, **16**, 647.

Bouchetal, la Roche, *Bull. soc. chim.*, 1903, **29**, 753; 1902, **27**, 451.

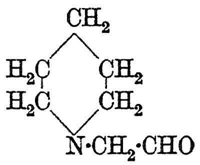
Piperidine-dicarboxylic Acid.

See Hexahydrocinchomeronic Acid *and* Hexahydrodipicolinic Acid.

Piperidinic Acid.

See 3-Amino-*n*-butyric Acid.

Piperidinoacetaldehyde



$C_7H_{13}ON$ MW, 127

Not known in free state.

B,HCl: cryst. from Et_2O. M.p. 103°. Decomp. on standing in air.

Chloroaurate: yellow cryst. M.p. 109–11°.

Chloroplatinate: orange-yellow needles. M.p. 121–2°.

Oxime: cryst. from EtOH–Et_2O. M.p. 135–6°.

Semicarbazone: cryst. M.p. 76°.

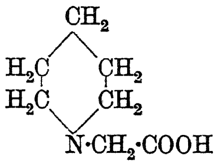
Di-Me acetal: $C_9H_{19}O_2N$. MW, 173. B.p. 94–6°/19 mm. *Methiodide*: m.p. 134·5–134·8°. *B,HCl*: 130–1°.

Di-Et acetal: $C_{11}H_{23}O_2N$. MW, 201. Liq. B.p. 219–21°. *B,HAuCl₄*: yellow plates. M.p. 96°. B_2,H_2PtCl_6: orange needles. M.p. 134°. *Methiodide*: cryst. M.p. 121°. *Ethiodide*: cryst. from C_6H_6. M.p. 123° (105°). *Picrate*: yellow needles. M.p. 67°.

Stoermer, *Ber.*, 1898, **31**, 2542.

Stoermer, Burkert, *Ber.*, 1894, **27**, 2016.

Piperidinoacetic Acid (*Piperidine-1-acetic acid, pentamethyleneglycine*)



$C_7H_{13}O_2N$ MW, 143

Prisms + $1H_2O$ from EtOH. M.p. anhyd. 215–17°. Sol. H_2O, EtOH, $CHCl_3$. Spar. sol. Et_2O, Me_2CO, ligroin, C_6H_6. Sublimes.

B,HCl: leaflets from EtOH–Et_2O. M.p. 215–16°.

Methochloride: m.p. 213° decomp.

Methochloroaurate: needles from H_2O. M.p. 178–9°.

Methochloroplatinate: orange cryst. M.p. 219°.

Me ester: $C_8H_{15}O_2N$. MW, 157. Oil. B.p. 205–7°.

Et ester: $C_9H_{17}O_2N$. MW, 171. Oil. B.p. 209°/732 mm. *B,HCl*: m.p. 130–1°. *Methochloride*: m.p. 189°. Hygroscopic. *Methiodide*: cryst. from EtOH. M.p. 158–9°. *Methochloroplatinate*: orange cryst. M.p. 225°.

Nitrile: $C_7H_{12}N_2$. MW, 124. Cryst. M.p. 19°. B.p. 210°, 99–100°/15 mm. *Methiodide*: leaflets. M.p. 192–3°.

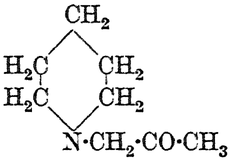
Ley, *Ber.*, 1909, **42**, 367.

Wedekind, *Ber.*, 1902, **35**, 182; 1899, **32**, 724.

Bischoff, *Ber.*, 1898, **31**, 2840.

Kraut, *Ann.*, 1871, **157**, 66.

Piperidinoacetone (N-*Acetonylpiperidine*)



$C_8H_{15}ON$ MW, 141

Liq. B.p. 195–7°. Misc. with H_2O and common org. solvents.

B,HAuCl$_4$: yellow plates. M.p. 107–8°.

B$_2$,H$_2$PtCl$_6$: orange prisms. M.p. 192–3°.

Methiodide: cryst. from EtOH. M.p. 126°.

Methochloroaurate: cryst. M.p. 85°.

Methochloroplatinate: orange cryst. from EtOH. M.p. 218–19°.

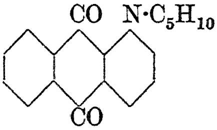
Oxime: needles from H_2O, plates from pet. ether. M.p. 104–5° (123°).

Phenylhydrazone: yellow leaflets from EtOH.Aq. M.p. 59–62°.

2 : 4-*Dinitrophenylhydrazone*: yellow needles from MeOH. M.p. 120–2°.

Matthaiopoulos, *Ber.*, 1898, **31**, 2398.
Stoermer, Burkert, *Ber.*, 1895, **28**, 1250.

1-Piperidinoanthraquinone



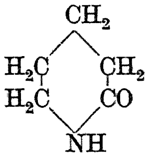
$C_{19}H_{17}O_2N$ MW, 291

Orange leaflets. M.p. 115°. Sol. conc. H_2SO_4 ⟶ yellow sol. Sol. $CHCl_3$–AcOH ⟶ purple sol.

Bayer, D.R.P., 136,777, (*Chem. Zentr.*, 1902, II, 1372).

Piperidinoethyl Alcohol.
See Hydroxyethylpiperidine.

α-Piperidone (*Valerolactam*, 2-*ketopiperidine*)



C_5H_9ON MW, 99

Hygroscopic cryst. M.p. 39–40°. B.p. 256°, 137°/14 mm., 64–5°/0·4 mm. Sol. EtOH, Et_2O, H_2O. Sol. dil. min. acids. Insol. alkalis.

B,HCl: cryst. from EtOH–Et_2O. M.p. 182–3°.

B,HgCl$_2$: needles + $1H_2O$. M.p. 187° decomp.

N-*Me*: $C_6H_{11}ON$. MW, 113. Hygroscopic liq. B.p. 104°/14 mm. D_{20}^{20} 1·0331. n_D^{20} 1·4818. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 104°. *B,HgCl$_2$*: needles + $1H_2O$. M.p. 119–20°.

N-*Et*: $C_7H_{13}ON$. MW, 127. Liq. B.p. 109°/12 mm. *B,HCl*: hygroscopic needles from EtOH–Et_2O. M.p. 108°. *B,HgCl$_2$*: needles + $1H_2O$. M.p. 113°.

N-*Propyl*: $C_8H_{15}ON$. MW, 141. Liq. B.p. 121°/14 mm. *B,HCl*: m.p. 112°.

N-*Isopropyl*: liq. B.p. 127–8°/15 mm. *B,HCl*: needles from EtOH–Et_2O. M.p. 118°. *B,HgCl$_2$*: m.p. 140–1°.

N-*Butyl*: $C_9H_{17}ON$. MW, 155. Liq. B.p. 130–1°/11 mm.

N-*Octyl*: $C_{13}H_{25}ON$. MW, 211. Liq. B.p. 172°/10 mm.

N-*Phenyl*: colourless needles from EtOH. M.p. 98°. *B,H$_2$PtCl$_6$*: reddish-brown cryst. M.p. 176°.

N-*Benzyl*: $C_{12}H_{15}ON$. MW, 189. Liq. B.p. 193°/8 mm.

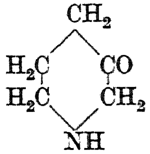
N-*Acetyl*: liq. B.p. 238°.

N-*Benzoyl*: leaflets from EtOH. M.p. 112°.

N-m-*Nitrobenzoyl*: yellow leaflets from EtOH.Aq. M.p. 114°.

Räth, *Ann.*, 1931, **489**, 111.
Ruzicka, *Helv. Chim. Acta*, 1921, **4**, 474.
Fischer, Zemplén, *Ber.*, 1909, **42**, 4886.
Fischer, Bergmann, *Ann.*, 1913, **398**, 114.
Wallach, *Ann.*, 1900, **312**, 179; 1902, **324**, 285.

β-Piperidone (3-*Ketopiperidine*)



C_5H_9ON MW, 99

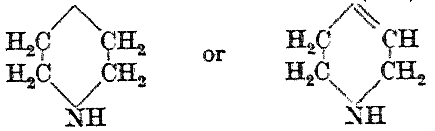
Not known in free state.

N-*Me*: $C_6H_{11}ON$. MW, 113. Liq. B.p. 63–4°/13 mm. D_{25}^{25} 0·9684. n_D^{25} 1·4459. Readily oxidised even by air. Reduces NH_3·$AgNO_3$ and Fehling's in the cold. *B,HCl*: cryst. from Me_2CO. M.p. 110–11°.

N-*Et*: *B,HCl*: cryst. + $1H_2O$ from MeOH–Et_2O. M.p. 172–173·5°.

McElwain, Voyze, *J. Am. Chem. Soc.*, 1949, **71**, 896.

γ-Piperidone (4-*Ketopiperidine*)



C_5H_9ON MW, 99

Yellow oil which cannot be distilled. Heat. ⟶ condensation products.

B,HCl: cryst. + $1\frac{1}{2}$EtOH from EtOH–Et_2O. M.p. 139–41°. M.p. (solvent free) 147–9°. Cryst. + $1H_2O$ from H_2O. M.p. 140–5° decomp.

N-*Me*: $C_6H_{11}ON$. MW, 113. Liq. with strong basic odour. B.p. 56–8°/8 mm. n_D^{25}

1·4588. *B,HCl*: cryst. from EtOH. M.p. 94·7–95·2°. *Methiodide*: cryst. + 1 MeOH from MeOH. M.p. 189–90°, m.p. (solvent free) 202–4° decomp. *Hydrogen oxalate*: colourless rods from EtOH–Et_2O. M.p. 122–3°.

N-*Et*: $C_7H_{13}ON$. MW, 127. B.p. 46–8°/1 mm. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 105–6°. *Methiodide*: pale yellow cryst. from isopropyl alcohol. M.p. 156–60°.

N-*Propyl*: $C_8H_{15}ON$. MW, 141. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 117–18°.

N-*Isopropyl*: b.p. 100–101°/27 mm. D_4^{25} 0·9495. n_D^{25} 1·4627.

N-*Butyl*: $C_9H_{17}ON$. MW, 155. B.p. 106–8°/22 mm. D_4^{25} 0·9292. n_D^{25} 1·4595. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 178–80°.

N-*Phenyl*: $C_{11}H_{13}ON$. MW, 175. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 145–7°.

N-*Benzyl*: $C_{12}H_{15}ON$. MW, 189. Liq. B.p. 114–16°/0·3 mm. D_{23}^{23} 1·0626. n_D^{23} 1·5374. *Semicarbazone*: m.p. 194–5°. *B,HCl*: cryst. from EtOH–Et_2O. M.p. 159–61°.

N-*Acetyl*: liq. B.p. 124–8°/0·2 mm. D_4^{25} 1·144. n_D^{25} 1·5023. Misc. with H_2O. Insol. Et_2O. 2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH. M.p. 212–13°.

N-*Benzoyl*: white cryst. M.p. 49–52°. B.p. 158–60°/0·2 mm. *Semicarbazone*: cryst. from AcOEt–EtOH. M.p. 193·5–195°.

N-*Nitroso*: pale yellow needles from C_6H_6-ligroin. M.p. 61–2°.

Oxime: white hygroscopic cryst. from C_6H_6. M.p. 117–18°.

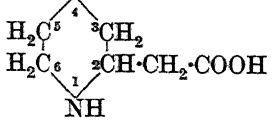
O-*Me*: b.p. 190·5–191°/738 mm. Volatile in steam. Strongly alkaline. *B,HgCl$_2$*: needles from H_2O. M.p. 191°.

O-*Et*: b.p. 96°/15 mm. Misc. with EtOH. Insol. H_2O.

O-*Propyl*: b.p. 218–20°/742 mm. *B,HCl*: needles from EtOH–Et_2O. M.p. 156–7°. *Picrate*: prisms from EtOH. M.p. 120–4°.

Kuettel, McElvain, *J. Am. Chem. Soc.*, 1931, **53**, 2696.
Bolyard, *J. Am. Chem. Soc.*, 1930, **52**, 1032.
McElvain, Bolyard, *J. Am. Chem. Soc.*, 1929, **51**, 924.
Ruzicka, Fornasir, *Helv. Chim. Acta*, 1920, **3**, 806.
Koenigs, Neumann, *Ber.*, 1915, **48**, 960.
McElwain, McMahon, *J. Am. Chem. Soc.*, 1949, **71**, 901.

2-Piperidylacetic Acid (2-*Piperidineacetic acid*)



$C_7H_{13}O_2N$ MW, 143

Needles from EtOH. M.p. 214°.

B,HCl: cryst. from EtOH. M.p. 180–2°.

B,HAuCl$_4$: yellow needles. M.p. 171–2° decomp.

Chloroplatinate: yellow cryst. powder. M.p. 203° decomp.

Königs, Happe, *Ber.*, 1903, **36**, 2906; 1902, **35**, 1348.

3-Piperidylacetic Acid (3-*Piperidineacetic acid*).

Me ester: N-*acetyl*, cryst. from MeOH–Me_2CO. M.p. 115–18°.

Et ester: $C_9H_{17}O_2N$. MW, 171. Liq. B.p. 101–3°/6 mm. D_4^{25} 1·0131. n_D^{25} 1·4643. *B_2,H_2PtCl$_6$*: m.p. 181°.

Nitrile: $C_7H_{12}N_2$. MW, 124. N-*Benzoyl*: Liq. B.p. 165–75°/0·1 mm. D_4^{20} 1·1056. n_D^{20} 1·5602.

N-*Me*: *picrate*, cryst. from MeOH. M.p. 112–13°. *Me ester*: b.p. 96°/13 mm.

Merchant, Marvel, *J. Am. Chem. Soc.*, 1928, **50**, 1199.
Hartmann, Bosshard, *Helv. Chim. Acta*, 1941, **24**, 33E.

4-Piperidylacetic Acid (4-*Piperidineacetic acid*).

White cryst. by pptn. from MeOH sol. with Me_2CO. M.p. 237–8° decomp.

Et ester: colourless liq. B.p. 123–7°/15 mm. *B,H$_2$PtCl$_6$*: cryst. from EtOH. M.p. 192° decomp.

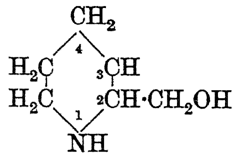
B,H$_2$PtCl$_6$: orange-red cryst. from H_2O. M.p. 210–13° decomp.

Prelog, Kohlbad, *Ann.*, 1937, **532**, 82.

2-Piperidylacetone.

See Isopelletierine.

α-Piperidylcarbinol (2-*Piperidylmethanol*, 2-*hydroxymethylpiperidine*)



$C_6H_{13}ON$ MW, 115

B.p. 80–3°/1 mm.

Picrate: m.p. 128–129·5°.

Renshaw *et al.*, *J. Am. Chem. Soc.*, 1939, **61**, 638.

β-Piperidylcarbinol (3-*Piperidylmethanol*, 3-*hydroxymethylpiperidine*).

Viscous oil. B.p. 106–7°/3·5 mm. D_4^{20} 1·0263. n_D^{20} 1·4964.

Sandborn, Marvel, *J. Am. Chem. Soc.*, 1928, **50**, 563.

γ-Piperidylcarbinol (4-*Piperidylmethanol*, 4-*hydroxymethylpiperidine*).

Hygroscopic cryst. M.p. 56–62°. B.p. 122°/12 mm., 118–20°/10 mm.

Picrate: yellow prisms from EtOH. M.p. 120°.

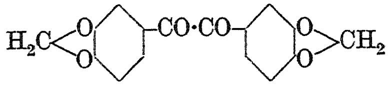
Clemo, Metcalfe, *J. Chem. Soc.*, 1937, 1523.
Rabe *et al.*, *J. prakt. Chem.*, 1938, **151**, 65.

Piperidylethylene.
See Vinylpiperidine.

4-[ω-4-Piperidylpropyl]-quinoline.
See Rubatoxan.

Piperidylpyridine.
See Pyridylpiperidine, Anabasine, Isonicotine *and* Isoneonicotine.

Piperil (3 : 4 : 3′ : 4′-*Dimethylenedioxy-benzil, dimethylene ether of veratril*)



$C_{16}H_{10}O_6$ MW, 298

Yellow needles from C_6H_6–EtOH. M.p. 171·5°. Sol. AcOH, $CHCl_3$, C_6H_6. Spar. sol. EtOH. Insol. ligroin. Alkalis ⟶ piperonylic acid.

Monoxime: cryst. from EtOH. M.p. 199°.

Dioxime: prisms from EtOH. M.p. 244° decomp.

Disemicarbazone: leaflets from AcOH. M.p. 250°.

Diphenylhydrazone: two forms. (i) Yellow needles from C_6H_6–EtOH. M.p. 183–4° decomp. (ii) Yellow needles. M.p. 219–20°.

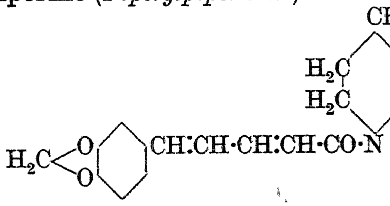
Di-o-*tolylhydrazone*: yellow cryst. from C_6H_6–EtOH. M.p. 206·5°.

Di-m-*tolylhydrazone*: yellow needles from C_6H_6–EtOH. M.p. 187°.

Di-p-*tolylhydrazone*: yellow needles from C_6H_6–EtOH. M.p. 215°.

Biltz, Arnd, *Ann.*, 1905, **339**, 272.
Biltz, Wienands, *Ann.*, 1899, **308**, 11.
Weiss, Appell, *J. Am. Chem. Soc.*, 1949, **71**, 2269.

Piperine (*Piperylpiperidine*)



$C_{17}H_{19}O_3N$ MW, 285

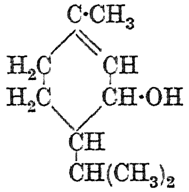
Constituent of pepper (*Piper nigrum, Piper longum, Piper Clusii*, etc.). Columns from EtOH, cryst. from C_6H_6–ligroin. M.p. 129·5°. Sol. AcOH, EtOH, $CHCl_3$, C_6H_6. Spar. sol. Et_2O. Insol. cold H_2O, pet. ether, dil. min. acids. Cryst. substance is prac. tasteless but alc. sols. have sharp burning taste. Alc. KOH ⟶ piperidine and piperic acid. Conc. H_2SO_4 ⟶ red col.

B,HBr: yellow cryst. powder. M.p. about 170°.

B_2,H_2SnBr_6: yellow cryst. M.p. 182–4° decomp.

Rügheimer, *Ber.*, 1882, **15**, 1391.
Cazeneuve, Caillot, *Bull. soc. chim.*, 1877, **27**, 291.
Dauber, *Ann.*, 1850, **74**, 204.
Peinemann, *Arch. pharm.*, 1896, **234**, 245.

Piperitol (Δ^1-p-*Menthenol*-3, 3-*carvomenthenol*, 1-*methyl*-4-*isopropylcyclohexenol*-3)



$C_{10}H_{18}O$ MW, 154

Constituent of essential oils of peppermint group of eucalyptus.

d-.

Liq. B.p. 165–70°/200 mm. D_{30}^{30} 0·911. n_D^{30} 1·474. $[\alpha]_D$ + 46·0°.

l-.

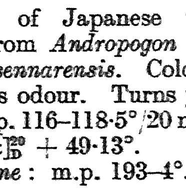
Liq. B.p. 95–6°/10 mm. D^{22} 0·923. n_D^{22} 1·476. $[\alpha]_D$ − 34·1°.

dl-.

Viscous oil with pleasant odour. B.p. 100–6°/19·5 mm. n_D^{18} 1·477.

Read, Walker, *J. Chem. Soc.*, 1934, 308.
Read, Storey, *J. Chem. Soc.*, 1930, 2772, 2779.

Piperitone (Δ^1-p-*Menthenone*-3, 1-*methyl*-4-*isopropylcyclohexenone*-3)



$C_{10}H_{16}O$ MW, 152

d-.

Constituent of Japanese peppermint oil, essential oil from *Andropogon Jwarancusa* and *Cymbopogon sennarensis*. Colourless oil with camphoraceous odour. Turns yellow on standing in air. B.p. 116–118·5°/20 mm. D_4^{20} 0·9344. n_D^{20} 1·4848. $[\alpha]_D^{20}$ + 49·13°.

Semicarbazone: m.p. 193–4°. $[\alpha]_D$ − 216·8°.

l-.

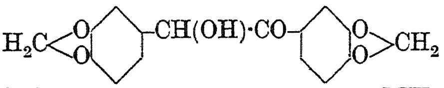
Occurs in essential oil of *Eucalyptus Dives*. B.p. 109·5–110·5°/15 mm. D_4^{20} 0·9324. n_D^{20} 1·4848. $[\alpha]_D^{20}$ − 51·53°.

Oxime: oil. $[\alpha]_D$ + 238·1°.

dl-.
Constituent of *Eucalyptus Dives* oil. B.p. 232–3°/769 mm., 113°/18 mm. D_4^{20} 0·9331. n_D^{20} 1·4845.
Oxime: two forms. (i) Cryst. from EtOH. M.p. 118–19°. (ii) M.p. 88–9°.
Semicarbazone: two forms. (i) M.p. 224–6°. (ii) M.p. 171–2°.

Walker, *J. Chem. Soc.*, 1935, 1585.
Read, Walker, *J. Chem. Soc.*, 1934, 308.
Howard, B.P. 410,813, (*Chem. Abstracts*, 1934, 6446).
Stephan, Düker, *J. prakt. Chem.*, 1931, **129**, 145.
Read, Smith, *J. Chem. Soc.*, 1923, **123**, 2268.
Simonsen, *J. Chem. Soc.*, 1921, **119**, 1646.

Piperoin (*Piperonyloin, piperonoin, 3 : 4 : 3′ : 4′-dimethylenedioxybenzoin*)



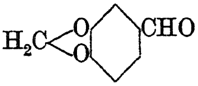
$C_{16}H_{12}O_6$ MW, 300

Needles from EtOH. M.p. 120°. Sol. EtOH, $CHCl_3$. Spar. sol. Et_2O. Fehling's ⟶ piperil.
Benzoyl: prisms from AcOH. M.p. 169°.
Oxalyl: crystals from $PhNO_2$. M.p. 169°.

Greene, Robinson, *J. Chem. Soc.*, 1922, **121**, 2187.
Smith, *Ann.*, 1896, **289**, 324.
Perkin, *J. Chem. Soc.*, 1891, **59**, 164.

2-Piperolidine.
See δ-Coniceine.

Piperonal (*Heliotropin, piperonyl aldehyde, 3 : 4-methylenedioxybenzaldehyde, methylene ether of protocatechuic aldehyde*)

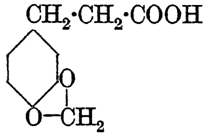


$C_8H_6O_3$ MW, 150

Occurs in *Robinia pseudoacacia*. Cryst. from H_2O. M.p. 37°. B.p. 263°, 185°/78 mm., 140°/15 mm.
$C_6H_3(NO_3)_2$*-1 : 3 : 5 add. comp.*: golden-yellow plates. M.p. 79°.
Oxime: *syn-*, cryst. from MeOH. M.p. 146°. *Acetyl*: m.p. 99°. *Benzoyl*: m.p. 168°. *Anti-*, needles from H_2O. M.p. 112°. *Acetyl*: m.p. 86°.
Di-Me acetal: $C_{10}H_{12}O_4$. MW, 196. Liq. B.p. 267–9°. D^{15} 1·206.
Di-Et acetal: $C_{12}H_{16}O_4$. MW, 224. Liq. B.p. 279–81°, 153–4°/11 mm. D^{15} 1·129.
Imide: $C_8H_7O_2N$. MW, 149. *B,HCl*: needles from C_6H_6. M.p. about 229–30°.
Methylimide: $C_9H_9O_2N$. MW, 163. M.p. 46°. B.p. 148°/16 mm. *B,HCl*: m.p. 220°.
Cyanhydrin: *see under* 3 : 4-Methylenedioxymandelic Acid.
Anil: piperonylideneaniline. $C_{14}H_{11}O_2N$. MW, 225. Needles from ligroin. M.p. 65°.
Diacetyl: cryst. from EtOH. M.p. 80° (51°).
Semicarbazone: leaflets. M.p. 237°.
Phenylhydrazone: needles from EtOH. M.p. 106°.
p-*Bromophenylhydrazone*: leaflets from EtOH, AcOH or C_6H_6. M.p. 155° decomp.
p-*Nitrophenylhydrazone*: red cryst. M.p. 199–200°.
2 : 4-*Dinitrophenylhydrazone*: purplish-red cryst. M.p. 265°.

McLang, *Chem. Abstracts*, 1927, **21**, 77.
Fritzsche, D.R.P., 207,702, (*Chem. Zentr.*, 1909, I, 1207).
Ciamician, Silber, *Ber.*, 1890, **23**, 1160.
Sherygin, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 975.

Piperonylacetic Acid (3 : 4-*Methylenedioxyhydrocinnamic acid*)

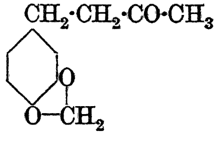


$C_{10}H_{10}O_4$ MW, 194

Needles from H_2O. M.p. 87–8°. B.p. 171–2°/11–12 mm. Red sol. in H_2SO_4. P_2O_5 ⟶ methylenedioxyhydrindone.
Et ester: $C_{12}H_{14}O_4$. MW, 222. Liq. B.p. 303°.
Chloride: $C_{10}H_9O_3Cl$. MW, 212·5. Yellow oil. Dist. under diminished pressure ⟶ 5 : 6-methylenedioxyhydrindone.
Amide: $C_{10}H_{11}O_3N$. MW, 193. Cryst. from H_2O or ligroin. M.p. 123·5°.
Methylamide: $C_{11}H_{13}O_3N$. MW, 207. Needles from Et_2O. M.p. 134°.
Anilide: needles. M.p. 122–3°.

Perkin, Robinson, *J. Chem. Soc.*, 1907, **91**, 1084.
Lorenz, *Ber.*, 1880, **13**, 758.
Regel, *Ber.*, 1887, **20**, 419.
Piccinini, *Chem. Zentr.*, 1904, I, 879.
Brochet, Bauer, *Bull. soc. chim.*, 1915, **17**, 52.
Decker, *Ann.*, 1913, **395**, 289.
Borsche, Eberlein, *Ber.*, 1914, **47**, 1470.
Dey, Govindachari, *Arch. Pharm.*, 1939, **277**, 177.

Piperonylacetone (3 : 4-*Methylenedioxybenzylacetone, methyl 3 : 4-methylenedioxyphenylethyl ketone, 3 : 4-methylenedioxy-1-γ-ketobutylbenzene*)



$C_{11}H_{12}O_3$ MW, 192

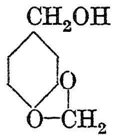
Leaflets from EtOH or pet. ether. M.p. 55°. B.p. 176·5°/15 mm., 164–5°/12 mm. Sol. most org. solvents.

Oxime: cryst. from EtOH or Et_2O. M.p. 100°.

Semicarbazone: needles. M.p. 166°.

Vavon, Faillebin, *Compt. rend.*, 1919, **169**, 66.
Kaufmann, Radosević, *Ber.*, 1916, **49**, 679.
Brochet, Cabaret, *Compt. rend.*, 1914, **159**, 328.
Gaind, Kapoor, Ray, *J. Indian Chem. Soc.*, 1941, **18**, 214.

Piperonyl Alcohol (3 : 4-*Methylenedioxybenzyl alcohol*)



$C_8H_8O_3$ MW, 152

Needles from pet. ether. M.p. 58°. B.p. 156–8°/15 mm. Sol. EtOH, Et_2O. Spar. sol. cold H_2O. Dist. ⟶ piperonal. Conc. H_2SO_4 ⟶ 2 : 3 : 6 : 7-dimethylenedioxy-9 : 10-dihydroanthracene.

Acetyl: two forms. (i) Cryst. from CCl_4 or pet. ether. M.p. 51°. (ii) Liq. B.p. 153–4°/14 mm. D_4^{18} 1·240. n_D^{18} 1·528.

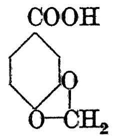
Benzoyl: needles from EtOH. M.p. 66°.

Phenylurethane: m.p. 102·5°.

Allophanate: m.p. 176·5° decomp.

Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1681.
Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 905.
Tiffeneau, Führer, *Bull. soc. chim.*, 1914, **15**, 172.
Barger, *J. Chem. Soc.*, 1908, **93**, 567.
Fittig, Remsen, *Ann.*, 1871, **159**, 130, 138.
Decker, Koch, *Ber.*, 1905, **38**, 1741.
Vavon, *Compt. rend.*, 1912, **154**, 361.
Palfray, Gauthier, *Bull. soc. chim.*, 1947, **48**, 678.

Piperonylic Acid (3 : 4-*Methylenedioxybenzoic acid*)



$C_8H_6O_4$ MW, 166

Needles from EtOH, cryst. from H_2O. M.p. 229°. Spar. sol. cold EtOH, Et_2O. Insol. cold H_2O, $CHCl_3$. Sublimes in prisms.

Me ester: $C_9H_8O_4$. MW, 180. Needles or leaflets from pet. ether. M.p. 53°. B.p. 273–4° (part. decomp.). Sol. MeOH, EtOH, Et_2O. Volatile in steam.

Et ester: $C_{10}H_{10}O_4$. MW, 194. Prisms. M.p. 18·5°. B.p. 285°. Sol. EtOH, Et_2O, pet. ether. Insol. H_2O.

Amide: $C_8H_7O_3N$. MW, 165. Prisms or needles from H_2O. M.p. 169°. Sol. EtOH, Et_2O. Spar. sol. H_2O. Insol. pet. ether.

Chloride: $C_8H_5O_3Cl$. MW, 184·5. Cryst. M.p. 80°. B.p. 155°/25 mm., 149–50°/12 mm.

Nitrile: $C_8H_5O_2N$. MW, 147. Needles from H_2O. M.p. 95°. Sol. EtOH, Et_2O, C_6H_6.

Anilide: cryst. from dil. EtOH. M.p. 146–7°.

Shriner, Kleiderer, *Organic Syntheses*, 1930, X, 82.
Barger, *J. Chem. Soc.*, 1908, **93**, 567.
Linge, *Rec. trav. chim.*, 1897, **16**, 47.
Rupe, Majewski, *Ber.*, 1900, **33**, 3403.

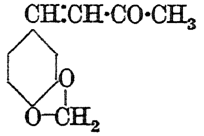
Piperonylidene-acetaldehyde.

See 3 : 4-Methylenedioxycinnamaldehyde.

Piperonylidene-acetic Acid.

See 3 : 4-Methylenedioxycinnamic Acid.

Piperonylideneacetone (*Methyl* 3 : 4-*methylenedioxystyryl ketone*, 3 : 4-*methylenedioxy*-1-γ-*keto*-α-*butenylbenzene*)



$C_{11}H_{10}O_3$ MW, 190

Cis:

Yellow cryst. M.p. 110–11°.

Trans:

Colourless cryst. M.p. 110–11°.

Sol. most org. solvents. Spar. sol. boiling H_2O. Volatile in steam.

Oxime: white cryst. from EtOH. M.p. about 186°.

Semicarbazone: two forms. (i) Given by *cis*- and *trans*-forms. Colourless cryst. from $CHCl_3$. M.p. 217°. Ultra-violet light ⟶ (ii). (ii) Yellow cryst. from C_6H_6–pet. ether. M.p. 168°.

Phenylhydrazone: prisms. M.p. about 166°.

Note.—The configuration of the above three derivatives is not known.

McGookin, Heilbron, *J. Chem. Soc.*, 1924, **125**, 2101.
Vavon, Faillebin, *Compt. rend.*, 1919, **169**, 67.
Wilson, Heilbron, Sutherland, *J. Chem. Soc.*, 1914, **105**, 2895.
Haber, *Ber.*, 1891, **24**, 620.
Nomura, Mozawa, *Chem. Abstracts*, 1919, **13**, 118.

ω-Piperonylideneacetophenone.

See 3 : 4-Methylenedioxychalkone.

Piperonylideneaniline.

See under Piperonal.

Piperonyloin.

See Piperoin.

Piperylene.
See 1 : 3-Pentadiene.

Piperylpiperidine.
See Piperine.

Pipitzahoic Acid.
See Perezone.

Pirylene.
See 1-Penten-3-yne.

Pivalic Acid (*Trimethylacetic acid*)

$(CH_3)_3C \cdot COOH$

$C_5H_{10}O_2$ MW, 102

M.p. 35·3–35·5°. B.p. 163·7–163·8°, 75–8°/20 mm. D^{50} 0·905.

Mercuric salt: needles from $CHCl_3$. M.p. 235°.

Me ester: $C_6H_{12}O_2$. MW, 116. B.p. 100–2°. D_4^0 0·891. n_D^{20} 1·3880.

Et ester: $C_7H_{14}O_2$. MW, 130. B.p. 118·5°. D^0 0·875. n_D^{20} 1·3912.

p-*Bromophenacyl ester*: m.p. 77–8°.

p-*Phenylphenacyl ester*: m.p. 113·1–114·0°.

1-*Naphthyl ester*: m.p. 66–66·5°.

Anhydride: $C_{10}H_{18}O_3$. MW, 186. B.p. 190°. n_D^{20} 1·4093.

Chloride: C_5H_9OCl. MW, 120·5. B.p. 105–6°.

Amide: $C_5H_{11}ON$. MW, 101. Needles or plates. M.p. 178°. B.p. about 212°.

Nitrile: C_5H_9N. MW, 83. Cryst. M.p. 15–16°. B.p. 105–6°.

Anilide: m.p. 127–9°.

Hardy, *J. Chem. Soc.*, 1936, 364–5.
du Pont, U.S.P., 1,995,930, (*Chem. Zentr.*, 1935, II, 594).
Sandborn, Bousquet, *Organic Syntheses*, 1928, VIII, 108.
Puntambeker, Zoellner, *ibid.*, 104.
Butlerow, *Ann.*, 1874, **173**, 355; 1873, **170**, 158.

Pivalic Aldehyde (*Trimethylacetaldehyde*)

$(CH_3)_3C \cdot CHO$

$C_5H_{10}O$ MW, 86

Constituent of wood spirit. Liq. Solidifies on cooling. M.p. 6°. B.p. 74–6°. D^{17} 0·7923. n_D^{20} 1·3791. Oxidises in air. Decomp. in daylight.

Trimer: $C_{15}H_{30}O_3$. MW, 258. Needles from EtOH. M.p. 82°.

Di-Et acetal: $C_9H_{20}O_2$. MW, 160. Liq. B.p. 146–8°/742 mm. D_4^{25} 0·8192. n_D 1·3942.

Oxime: m.p. 41°. B.p. 65°/20 mm.

Semicarbazone: m.p. 191°.

2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH. M.p. 210°.

Anil: b.p. 101–2°/20 mm.

Azine: m.p. 79°.

Dunbar, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 444.
Conant, Webb, Mendum, *J. Am. Chem. Soc.*, 1929, **51**, 1250.
Campbell, *J. Am. Chem. Soc.*, 1937, **59**, 1982.

Pivalone (*Hexamethylacetone, di-*tert.*-butyl ketone, 2 : 2 : 4 : 4-tetramethylpentanone-3*)

$(CH_3)_3C \cdot CO \cdot C(CH_3)_3$

$C_9H_{18}O$ MW, 142

Liq. B.p. 152°, 70°/43 mm. D^{18} 0·824. n_D^{18} 1·4195. Does not react with usual ketonic reagents.

Vavon, Ivanoff, *Compt. rend.*, 1923, **177**, 453.
Haller, Bauer, *Compt. rend.*, 1910, **150**, 584.
Henderson, Henderson, Heilbron, *Ber.*, 1914, **47**, 887.

Pivalophenone.
See *tert.*-Butyl phenyl Ketone.

Plasmoquin.
See Pamaquin.

Platyphylline

$C_{18}H_{25}O_5N$ MW, 335

Alkaloid from *Senecio platyphyllus*, D.C. Needles from H_2O. M.p. 124–5°. Very sol. $CHCl_3$. Sol. EtOH, Me_2CO, Et_2O, C_6H_6. Spar. sol. hot H_2O, pet. ether. $[\alpha]_D$ — 45·09°.

Perchlorate: prisms from H_2O. M.p. 222–3° decomp.

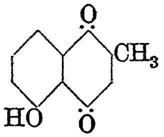
B,HAuCl$_4$: yellow needles + $\frac{1}{2}H_2O$ from EtOH.Aq. M.p. 200–1° decomp.

Methiodide: needles from EtOH. M.p. 216–17°. $[\alpha]_D$ — 31·27°.

Picrolonate: dark yellow needles from EtOH. M.p. 205–6° decomp.

Orechoff, Konowalowa, Tiedebel, *Ber.*, 1935, **68**, 1886.
Orechoff, *ibid.*, 653.
Orechoff, Tiedebel, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 273, (*Chem. Abstracts*, 1938, **32**, 5403).

Plumbagin (*5-Hydroxy-2-methyl-*1 : 4-*naphthoquinone*)



$C_{11}H_8O_3$ MW, 188

Constituent of various species of *Plumbago*. Yellow needles from EtOH.Aq. M.p. 78–9°. Sol. Et_2O, Me_2CO, AcOH, $CHCl_3$, C_6H_6. Insol. cold H_2O. Volatile in steam. Sublimes.

Dioxime: yellow needles from EtOH. M.p. 220°.

Semicarbazone: light brown needles from Py–EtOH. M.p. above 280°.

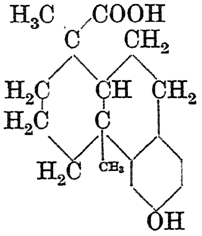
Phenylhydrazone: reddish-brown needles from EtOH. M.p. 198°.

Acetyl: yellow needles. M.p. 117–18° (138°).

Benzoyl: yellow prisms from EtOH.Aq. M.p. 147°.

Fieser, Dunn, *J. Am. Chem. Soc.*, 1936, **58**, 572.
Buruaga, Verdú, *Chem. Zentr.*, 1935, I, 3146.
Roy, Dutt, *J. Indian Chem. Soc.*, 1928, **5**, 419.
Dieterle, *Chem. Abstracts*, 1939, **33**, 2891.

Podocarpic Acid (6-*Hydroxy*-1 : 4*a*-*dimethyl*-1 : 2 : 3 : 4 : 4*a* : 9 : 10 : 10*a*-*octahydrophenanthrene*-1-*carboxylic acid*)



$C_{17}H_{22}O_3$ MW, 274

Occurs in *Podocarpus cupressina, var. imbricata*. Plates from EtOH.Aq. M.p. 193·5°. Sol. EtOH, Et_2O, AcOH. Insol. H_2O, $CHCl_3$, CS_2, C_6H_6. $[\alpha]_{578}$ + 144° in EtOH.

Me ester: $C_{18}H_{24}O_3$. MW, 288. Cryst. from EtOH. M.p. 208°. *Me ether*: cryst. from pet. ether. M.p. 128°. *Benzoyl*: rods from MeOH. M.p. 143–5°.

Et ester: $C_{19}H_{26}O_2$. MW, 302. Needles from EtOH. M.p. 161°.

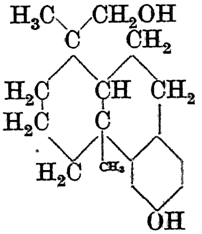
p-*Nitrobenzyl ester*: m.p. 204°.

Me ether: $C_{18}H_{24}O_3$. MW, 288. M.p. 157–8°.

Acetyl: prisms from Et_2O. M.p. 173–6°.

Sherwood, Short, *Chem. Abstracts*, 1934, **28**, 6435.
Oudemans, *Ann.*, 1873, **170**, 214.
Sherwood, Short, *J. Chem. Soc.*, 1938, 1006.
Campbell, Todd, *J. Am. Chem. Soc.*, 1942, **64**, 928.
Haworth, Moore, *J. Chem. Soc.*, 1946, 633.

Podocarpinol

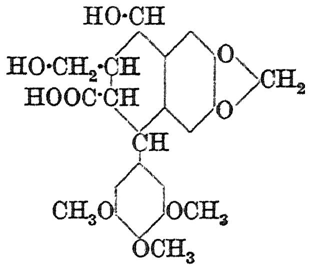


$C_{17}H_{24}O_2$ MW, 260

Cubes from Et_2O–hexane. M.p. 178–9°.

Zeiss, Slimowicz, Pasternak, *J. Am. Chem. Soc.*, 1948, **70**, 1981.

Podophyllic Acid



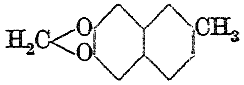
$C_{22}H_{24}O_9$ MW, 432

Needles from $CHCl_3$ or EtOH–C_6H_6. M.p. 163–5°. Sol. EtOH. Spar. sol. H_2O, Et_2O, AcOEt, $CHCl_3$, CCl_4, C_6H_6. $[\alpha]_D^{16}$ −102·8° in EtOH.

Hydrazide: plates from H_2O. M.p. 155–60°.

Borsche, Niemann, *Ann.*, 1932, **494**, 136.
Mellanoff, Schaeffer, *Chem. Zentr.*, 1927, II, 1589.

Podophyllomerol (6 : 7-*Methylenedioxy*-2-*methylnaphthalene*)



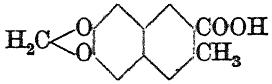
$C_{12}H_{10}O_2$ MW, 186

Plates from MeOH. M.p. 129–129·5°.

Picrate: orange-red needles. M.p. 133–4°.

Borsche, Niemann, *Ann.*, 1932, **499**, 67.

Podophyllomeronic Acid (*Methylene ether of phyllomeronic acid*, 6 : 7-*methylenedioxy*-3-*methyl*-2-*naphthoic acid*)



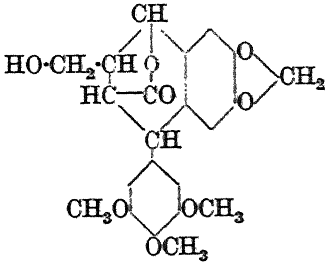
$C_{13}H_{10}O_4$ MW, 230

Plates from AcOH. M.p. 239–40°.

Me ester: $C_{14}H_{12}O_4$. MW, 244. Needles from MeOH. M.p. 125–6°.

Borsche, Niemann, *Ann.*, 1932, **494**, 141; 1932, **499**, 67.
Späth, Wesselý, Kornfeld, *Ber.*, 1932, **65**, 1547.

Podophyllotoxin



$C_{22}H_{22}O_8$ MW, 414

Occurs in *Podophyllum*. Needles from Me_2CO.Aq. or MeOH. M.p. 114–18°. (M.p. anhyd. 157°.) Sol. EtOH, Me_2CO, AcOH, $CHCl_3$, hot C_6H_6. Spar. sol. EtOH.Aq. Insol. H_2O, Et_2O, ligroin, C_6H_6. $[\alpha]_D^{14}$ −101·3° in EtOH.

Acetyl: needles from MeOH. M.p. 204°. $[\alpha]_D^{16}$ −134·9° in $CHCl_3$.

Hartwell *et al.*, *J. Am. Chem. Soc.*, 1953, **75**, 235.
Späth *et al.*, *Ber.*, 1932, **65**, 1542.
Borsche, Niemann, *Ann.*, 1932, **494**, 131; 1932, **499**, 63.
Mellanoff, Schaeffer, *Chem. Zentr.*, 1927, II, 1589.

μ-Polychloroprene.
See under Chloroprene.

Polygalitol.
See 1 : 5-Anhydro-D-sorbitol.

Polyoxymethylene.
See under Formaldehyde.

Polyporenic Acid A

$C_{31}H_{50}O_4$ MW, 486

Isolated from the fungus, *Polyporus betulinus*, Fr. Needles from MeOH.Aq., Me_2CO.Aq. or dil. AcOH. M.p. 199–200°. Spar. sol. Et_2O, C_6H_6, $CHCl_3$. $[\alpha]_D^{20}$ + 69° in Py. Tetranitromethane in $CHCl_3$ ⟶ yellow col. H_2SO_4–$CHCl_3$ ⟶ red col. Liebermann–Burchard reagent ⟶ red col. with green fluor.

Me ester: $C_{32}H_{52}O_4$. MW, 500. Needles from MeOH. M.p. 148–9°. $[\alpha]_D^{20}$ + 79·5° in $CHCl_3$. Contains 2 active H atoms and 2 ethylenic linkages. *Monoformyl*: plates from MeOH. M.p. 148°. $[\alpha]_D^{20}$ + 84° in $CHCl_3$. *Monoacetyl*: needles from EtOH.Aq. M.p. 137–8°. $[\alpha]_D^{20}$ + 33° in $CHCl_3$. *Monobenzoyl*: needles from MeOH. M.p. 142–3°. $[\alpha]_D^{20}$ + 12·5° in $CHCl_3$. *Diacetyl*: needles from MeOH. M.p. 118–20°. $[\alpha]_D^{20}$ + 86° in $CHCl_3$.

Cross, Eliot, Heilbron, Jones, *J. Chem. Soc.*, 1940, 632.
Cross, Jones, *ibid.*, 1491.
Curtis, Heilbron, Jones, Woods, *J. Chem. Soc.*, 1953, 457.
Jones, Woods, *ibid.*, 464.
Halsall, Jones, Lemin, *ibid.*, 468.

Polyporenic Acid B

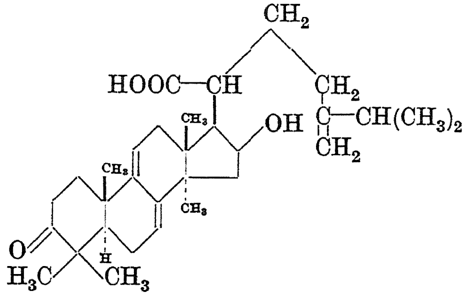
$C_{31}H_{50}O_4$ MW, 486

Constituent of the fungus, *Polyporus betulinus*, Fr. Asbestos-like mass from Me_2CO or AcOH. M.p. 300–10° decomp. Spar. sol. cold Py. Prac. insol. Et_2O, C_6H_6, $CHCl_3$. Tetranitromethane in $CHCl_3$ ⟶ yellow col. H_2SO_4–$CHCl_3$ ⟶ orange col. Liebermann–Burchard reagent ⟶ violet col.

Me ester: Needles from MeOH.Aq. Softens at 155°, m.p. 160°. Contains 2 active H atoms and 2 ethylenic linkages.

Cross, Eliot, Heilbron, Jones, *J. Chem. Soc.*, 1940, 632.

Polyporenic Acid C



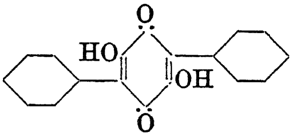
$C_{31}H_{46}O_4$ MW, 482

Constituent of the fungi, *Polyporus betulinus*, Fr., and *Polyporus benzoinus* (Wahl.) Fr. M.p. 273–6°. $[\alpha]_D^{20}$ + 6° in Py. ($[\alpha]_D^{20}$ + 8·2° in Py.).

Me ester: m.p. 198–9°. $[\alpha]_D^{20}$ + 10°. *Acetyl*: m.p. 122–4°. $[\alpha]_D^{20}$ − 29°. 2 : 4-*Dinitrophenylhydrazone*: m.p. 195–8°.

Cross, Eliot, Heilbron, Jones, *J. Chem. Soc.*, 1940, 632.
Cross, Jones, *ibid.*, 1491.
Birkinshaw, Morgan, Findlay, *Biochem. J.*, 1952, **50**, 509.
Bowers, Halsall, Jones, Lemin, *J. Chem. Soc.*, 1953, in the Press.

Polyporic Acid (3 : 6-*Dihydroxy*-2 : 5-*diphenyl*-p-*benzoquinone*)



$C_{18}H_{12}O_4$ MW, 292

Occurs in *Polyporus*. Bronze plates from toluene. M.p. 305°. Spar. sol. EtOH, $CHCl_3$. Insol. H_2O, Et_2O, CS_2, C_6H_6. Sublimes.

Di-Me ether: $C_{20}H_{16}O_4$. MW, 320. Red cryst. from EtOH. M.p. 187°.

Di-Et ether: $C_{22}H_{20}O_4$. MW, 348. Yellow needles or orange-red prisms from EtOH. M.p. 134°.

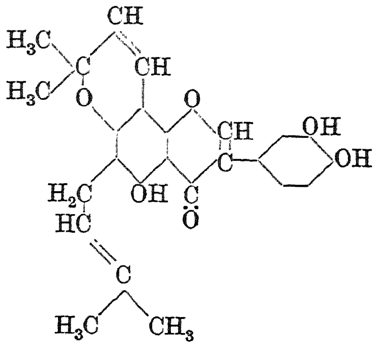
Diacetyl: needles from *n*-butyl alcohol. M.p. 246° (209°).

Shildneck, Adams, *J. Am. Chem. Soc.*, 1931, **53**, 2377.
Kögl, *Ann.*, 1926, **447**, 78.
Stahlschmidt, *Ann.*, 1877, **187**, 177.

Polystichocitrin.
See Flavaspidic Acid.

Polythene.
See under Ethylene.

Pomiferin



$C_{25}H_{24}O_6$ MW, 420

Pigment from the fruit of the osage orange tree (*Maclura pomifera*, Raf.). Yellow cryst. from xylene, C_6H_6 or EtOH. M.p. 200·5°.

Di-Me ether: $C_{27}H_{28}O_6$. MW, 448. Yellow needles from EtOH. M.p. 132°. *Monoacetyl*: needles from EtOH. M.p. 128–9°.

Tri-Me ether: $C_{28}H_{30}O_6$. MW, 462. Cryst. from EtOH. M.p. 139·5°.

Diacetyl: yellow plates from MeOH.Aq. M.p. 134·5°.

Triacetyl: needles from EtOH or C_6H_6–pet. ether. M.p. 154°.

*Di-*p*-toluenesulphonyl*: cryst. from EtOH. M.p. 148°.

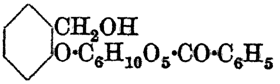
Wolfrom, Benton, Gregory, Hess, Mahan, Morgan, *J. Am. Chem. Soc.*, 1939, **61**, 2833.
Wolfrom, Gregory, *J. Am. Chem. Soc.*, 1940, **62**, 651.
Wolfrom, Mahan, *J. Am. Chem. Soc.*, 1941, **63**, 1253.
Wolfrom *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 406.

Pongamol.

Yellow rhombic prisms from EtOH–AcOH. M.p. 128–9°. Mod. sol. AcOH. Slightly sol. EtOH. Insol. H_2O. EtOH sol. with $FeCl_3 \longrightarrow$ intense red col.

Rangaswami, Seshadri, *Chem. Abstracts*, 1941, **35**, 5733.

Populin (6-*Benzoylsalicin*)



$C_{20}H_{22}O_8$ MW, 390

Occurs in leaves and bark of *Populus tremula* and in buds of *P. nigra*, *P. pyramidalis*, etc. Needles + $2H_2O$ from H_2O, prisms from EtOH. M.p. 180°. Sol. hot H_2O, EtOH. Spar. sol. cold H_2O. $[\alpha]_D$ −53°, −2·0° in Py. Conc. $H_2SO_4 \longrightarrow$ red col. $Ba(OH)_2 \longrightarrow$ salicin.

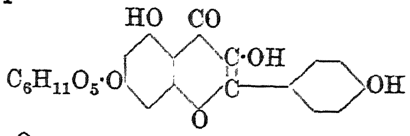
Tetra-Me ether: $C_{24}H_{30}O_8$. MW, 446. Needles from Et_2O–ligroin. M.p. 134–5°. $[\alpha]_D$ −31·7° in $CHCl_3$.

Richtinger, Yeakel, *J. Am. Chem. Soc.*, 1934, **56**, 2495.

Populnetin.

See Kaempferol.

Populnin

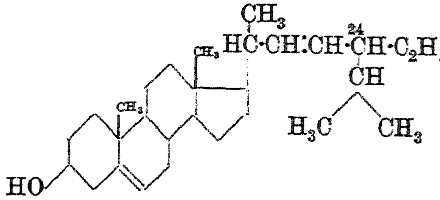


$C_{21}H_{20}O_{11}$ MW, 448

Pigment isolated from Indian tulip flowers. M.p. 228–30° decomp. Hyd. $\longrightarrow$ glucose + kaempferol (populnetin). $FeCl_3 \longrightarrow$ pale green col.

Rao, Seshadri, *Chem. Abstracts*, 1947, **41**, 3798.

Poriferasterol (C_{24} *Epimer of stigmasterol*)



$C_{29}H_{48}O$ MW, 412

Isolated from certain sponges. Cryst. from EtOH, Et_2O. M.p. 156°. $[\alpha]_D$ − 46° in $CHCl_3$.

3β-*Acetyl*: m.p. 146·5–147°. $[\alpha]_D$ − 53° in $CHCl_3$.

3β-*Benzoyl*: m.p. 139·5–140·5°. $[\alpha]_D$ − 22° in $CHCl_3$.

3β-*Acetyl dibromide*: m.p. 211–12°. $[\alpha]_D$ − 31° in $CHCl_3$.

3β-*Acetyl tetrabromide*: m.p. 185° decomp. $[\alpha]_D$ − 43·5° in $CHCl_3$.

Tetrahydro deriv.: m.p. 143–4°. $[\alpha]_D$ + 25° in $CHCl_3$. *Acetyl*: m.p. 140–1°. $[\alpha]_D$ + 16·3° in $CHCl_3$.

Valentine, Bergmann, *J. Org. Chem.*, 1941, **6**, 452.
Lyon, Bergmann, *J. Org. Chem.*, 1942, **7**, 428.
Bergmann, Low, *J. Org. Chem.*, 1947, **12**, 67.

Porphyroxine.

$C_{19}H_{23}O_4N$ MW, 329

Prisms from ligroin. M.p. 134–5°. Sol. $CHCl_3$, Me_2CO, AcOH, CS_2. Mod. sol. MeOH, AcOEt, CCl_4, C_6H_6. Spar. sol. H_2O. $[\alpha]^{32}$ −139·9° in $CHCl_3$.

B,HCl: needles from H_2O. M.p. 155°. $[\alpha]^{32}$ −118·8° in H_2O.

B,HBr: needles. M.p. 148–50°. $[\alpha]^{32}$ −90·6° in H_2O.

B,HI: decomp. at 115°. $[\alpha]^{32}$ −77·8° in H_2O.

B_2,H_2SO_4: plates from H_2O. M.p. 193°.

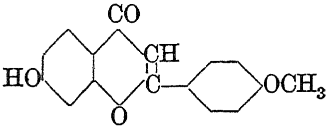
Methiodide: m.p. 150–2°.
Methosulphate: needles from MeOH. M.p. 205° decomp.
Oxime: m.p. 198° decomp.
Phenylhydrazone: m.p. 150° decomp.
Semicarbazone: decomp. at 244°.
Acetyl: m.p. 125°. *B,HCl*: needles. M.p. 126° decomp. *B,HBr*: m.p. 155° decomp. B_2,H_2SO_4: m.p. 190° decomp.
Me ether: $C_{20}H_{25}O_4N$. MW, 343. Cryst. from pet. ether. M.p. 125–6°. *Oxime*: needles from MeOH. M.p. 185–6°. *Phenylhydrazone*: m.p. 189° decomp. *Semicarbazone*: cryst. from EtOH. M.p. 217°.

Rakshit, *J. Chem. Soc.*, 1919, **115**, 455; *Ber.*, 1926, **59**, 2473.
Machiguchi, *J. Pharm. Soc. Japan*, 1926, No. **529**, 185, (*Chem. Abstracts*, 1926, **20**, 2725).
Henry, *The Plant Alkaloids*, 4th Ed., (J. & A. Churchill Ltd., London), 220.

It has been suggested that porphyroxine is impure codeine.

Rajagopalan, *J. Org. Chem.*, 1945, **10**, 175.

Pratol (7-*Hydroxy*-4′-*methoxyflavone*)

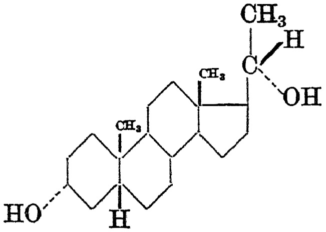


$C_{16}H_{12}O_4$ MW, 268

Occurs in flowers of *Trifolium* species. Pale yellow needles from EtOH. M.p. 263–4°. Sol. hot EtOH. Spar. sol. H_2O, Et_2O, $CHCl_3$, C_6H_6.
Acetyl: needles or hexagonal plates from EtOH. M.p. 176–7°.

Baker, *J. Chem. Soc.*, 1933, 1386.
Power, Salway, *J. Chem. Soc.*, 1910, **97**, 233, 1008.
Nadkarni, Wheeler, *J. Chem. Soc.*, 1938, 1321.

Pregnan-3α : 20α-diol ("*Pregnanediol*")

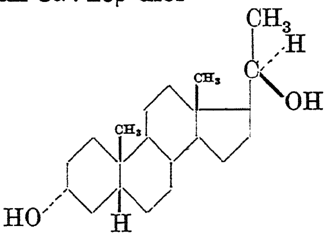


$C_{21}H_{36}O_2$ MW, 320

Isolated from urine of pregnancy. Plates from Me_2CO. M.p. 243–4°. Spar. sol. most org. solvents. Gives none of the recognised sterol colour reactions.
3-*Acetyl*: cryst. from 85% MeOH. M.p. 132–5° (retains solvent).
20-*Acetyl*: cryst. from dil. Me_2CO. M.p. 174–175·5°.
Diacetyl: cryst. from MeOH. Melts first at 168°, solidifies and melts finally at 182–3°.

Butenandt, Schmidt, *Ber.*, 1934, **67**, 1895.
Butenandt, Hildebrandt, Brücher, *Ber.*, 1931, **64**, 2529.
Butenandt, *Ber.*, 1930, **63**, 661.
Marrian, *Biochem. J.*, 1929, **23**, 1090.
Marker, Rohrmann, *J. Am. Chem. Soc.*, 1940, **62**, 518.

Pregnan-3α : 20β-diol

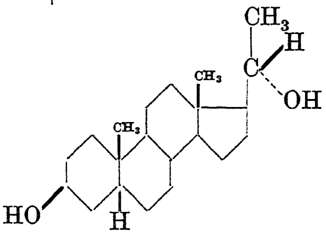


$C_{21}H_{36}O_2$ MW, 320

Cryst. from EtOH. M.p. 244–6°.
Diacetyl: cryst. from MeOH. M.p. 113–15°.

Meystre, Miescher, *Helv. Chim. Acta*, 1946, **29**, 47.

Pregnan-3β : 20α-diol

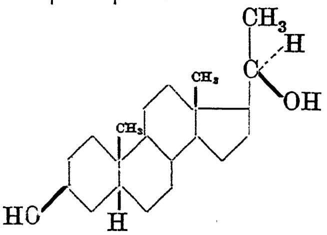


$C_{21}H_{36}O_2$ MW, 320

Cryst. from Me_2CO. M.p. 182°.
Diacetyl: cryst. from EtOH.Aq. M.p. 141°.
Dibenzoyl: plates from EtOH. M.p. 167–8°.

Pearlman, *J. Am. Chem. Soc.*, 1944, **66**, 806.
Marker *et al.*, *J. Am. Chem. Soc.*, 1937, **59**, 2295.

Pregnan-3β : 20β-diol

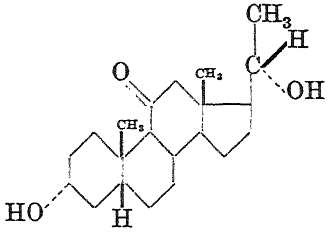


$C_{21}H_{36}O_2$ MW, 320

Cryst. from EtOH.Aq. M.p. 174–6°.

Marker, Rohrmann, *J. Am. Chem. Soc.*, 1940, **62**, 518.

Pregnan-3α : 20α-diol-11-one



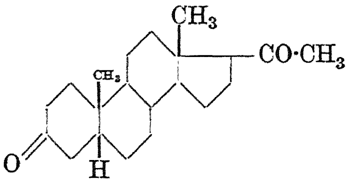
$C_{21}H_{34}O_3$ MW, 334

Isolated from urine of male and female Addisonians. Cryst. from $MeOH-Me_2CO$. M.p. 217–19°. $[\alpha]_D$ + 59° in EtOH.

3 : 20-*Diacetyl* : m.p. 233°.

Mason, *J. Biol. Chem.*, 1948, **172**, 783.

Pregnan-3 : 20-dione



$C_{21}H_{32}O_2$ MW, 316

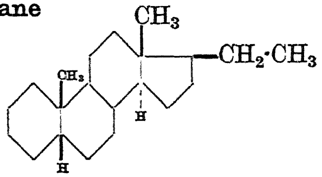
Occurs in human pregnancy urine and mares pregnancy urine. Cryst. from Me_2CO.Aq. M.p. 123°.

Bis-dinitrophenylhydrazone : m.p. 207–11°.

Lieberman *et al.*, *J. Biol. Chem.*, 1948, **172**, 263.

Marker *et al.*, *J. Am. Chem. Soc.*, 1938. **60**, 1559.

Pregnane

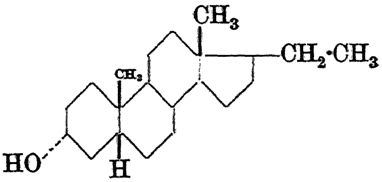


$C_{21}H_{36}$ MW, 288

Cryst. from MeOH. M.p. 83·5°. $[\alpha]_D^{20}$ + 21·2° in $CHCl_3$.

Butenandt, Hildebrandt, Brücher, *Ber.*, 1931, **64**, 2538.

Pregnan-3α-ol



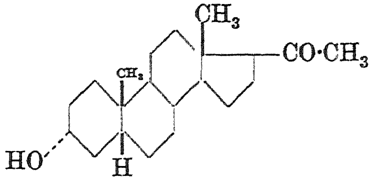
$C_{21}H_{36}O$ MW, 304

Occurs in human pregnancy urine. Cryst. from MeOH.Aq. M.p. 148°.

Acetyl : m.p. 106°.

Marker, Lawson, *J. Am. Chem. Soc.*, 1938, **60**, 2638, 2928.

Pregnan-3α-ol-20-one



$C_{21}H_{34}O_2$ MW, 318

Isolated from urine of pregnant women and sows. Long needles from C_6H_6. M.p. 148·5–149·5°. $[\alpha]_D$ + 107° in EtOH.

Acetyl : m.p. 94–7°.

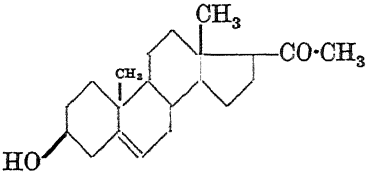
Lieberman *et al.*, *J. Biol. Chem.*, 1948, **172**, 263.

Sutherland, Marrian, *Biochem. J.*, 1946, **40**, lxi.

Marker, Kamm, *J. Am. Chem. Soc.*, 1937, **59**, 1373.

Marker, Rohrmann, *J. Am. Chem. Soc.*, 1939, **61**, 3476.

Δ⁵-Pregnen-3β-ol-20-one ("*Pregnenolone*")



$C_{21}H_{32}O_2$ MW, 316

Needles from MeOH. M.p. 189–90°. $[\alpha]_D$ + 30° in EtOH.

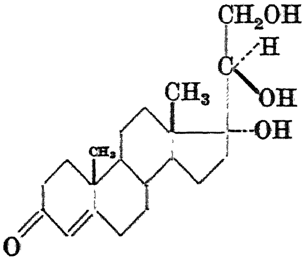
Acetyl : m.p. 146–7°. $[\alpha]_D$ + 22° in EtOH.

Semicarbazone : m.p. 262–3° decomp.

Butenandt, Westphal, Cobler, *Ber.*, 1934, **67**, 1611.

Fieser, Huang-Minlon, *J. Am. Chem. Soc.*, 1949, **71**, 1840.

Δ⁴-Pregnen-17α : 20β : 21-triol-3-one



$C_{21}H_{32}O_4$ MW, 348

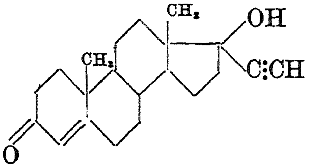
Cryst. from MeOH. M.p. 190°. $[\alpha]_D$ + 63° in dioxan.

20 : 21-*Diacetyl* : m.p. 189–90°. $[\alpha]_D$ + 125° in dioxan.

Ruzicka, Müller, *Helv. Chim. Acta*, 1939, **22**, 755.

Logemann, *Naturwiss.*, 1939, **27**, 196.

Pregnenynolone (17-*Ethynyltestosterone*)



$C_{21}H_{28}O_2$ MW, 312

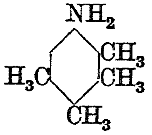
Cryst. from $CHCl_3$-Me_2CO or AcOEt. M.p. 270–2° corr. $[\alpha]_D^{20}$ + 22·5° in dioxan. Absorption maximum at 2385 Å. More effective than progesterone for oral administration.

Oxime: cryst. from EtOH.Aq. M.p. 234–5° decomp.

Semicarbazone: cryst. from Me_2CO. M.p. 230–1° decomp.

Inhoffen, *Ber.*, 1939, **72**, 595.
Ruzicka, Hofmann, Meldahl, *Helv. Chim. Acta*, 1938, **21**, 371.
Ruzicka, Hofmann, *Helv. Chim. Acta*, 1937, 20, 1280.

Prehnidine (2 : 3 : 4 : 5-*Tetramethylaniline*, 5-*amino*-1 : 2 : 3 : 4-*tetramethylbenzene*, 5-*amino-prehnitene*)



$C_{10}H_{15}N$ MW, 149

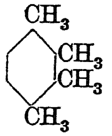
Leaflets from H_2O. M.p. 70° (64–6°). B.p. 259–60°. Sol. EtOH, Et_2O, pet. ether. Mod. sol. hot H_2O.

N-*Formyl*: needles from H_2O. M.p. 143–4°.

N-*Acetyl*: needles from EtOH.Aq. M.p. 172° (169·5°).

Limpach, *Ber.*, 1888, **21**, 644.
Töhl, *ibid.*, 905.

Prehnitene (*Prehnitol*, 1 : 2 : 3 : 4-*tetramethylbenzene*)



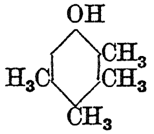
$C_{10}H_{14}$ MW, 134

Liq. Solidifies on cooling. M.p. − 6·4°. B.p. 203–4°, 96·4°/25 mm., 75–75·5°/6·5 mm. D_4^{20} 0·901. n_D^{25} 1·5196.

Picrate: yellow prisms from EtOH. M.p. 92–5°.

Smith, Cass, *J. Am. Chem. Soc.*, 1932, **54**, 1620.
Baril, Hauber, *J. Am. Chem. Soc.*, 1931, **53**, 1089.
Birch, Dean, Fidler, Lowry, *J. Am. Chem. Soc.*, 1949, **71**, 1364.

Prehnitenol (2 : 3 : 4 : 5-*Tetramethylphenol*, 5-*hydroxyprehnitene*, 5-*hydroxy*-1 : 2 : 3 : 4-*tetramethylbenzene*)



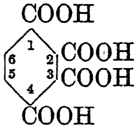
$C_{10}H_{14}O$ MW, 150

Needles from EtOH.Aq. or ligroin. M.p. 86–7°. B.p. 266°. Very sol. EtOH, Et_2O. Spar. sol. H_2O, ligroin. Volatile in steam.

Acetyl: prisms from ligroin. M.p. 56–7°.

Bamberger, Blangey, *Ann.*, 1911, **384**, 307.
Töhl, *Ber.*, 1888, **21**, 907.

Prehnitic Acid (*Benzene*-1 : 2 : 3 : 4-*tetracarboxylic acid*)



$C_{10}H_6O_8$ MW, 254

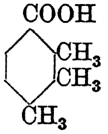
Prisms from H_2O. M.p. 238°.

1 : 4-*Di-Me ester*: $C_{12}H_{10}O_8$. MW, 282. M.p. 176–7°.

Tetra-Me ester: $C_{14}H_{14}O_8$. MW, 310. M.p. 133–5°.

Smith, Byrkit, *J. Am. Chem. Soc.*, 1933, **55**, 4305.
Doebner, *Ann.*, 1900, **311**, 143.
Baeyer, *Ann.*, 1873, **166**, 325.
Meyer, Sudborough, *Ber.*, 1894, **27**, 1591.
Smith, Carlson, *J. Am. Chem. Soc.*, 1939, **61**, 288.

Prehnitylic Acid (2 : 3 : 4-*Trimethylbenzoic acid*)

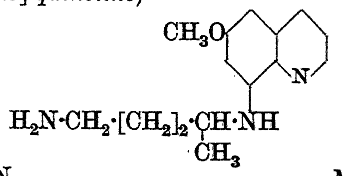


$C_{10}H_{12}O_2$ MW, 164

Prisms from EtOH. M.p. 167·5°. $k = 3{\cdot}49 \times 10^{-5}$ at 25°. Mod. volatile in steam.

Jacobsen, *Ber.*, 1886, **19**, 1214.
Lapworth, Chapman, *J. Chem. Soc.*, 1900, **77**, 316.

Primaquine (6-*Methoxy*-8-[4-*amino*-1-*methylbutylamino*]-*quinoline*)



$C_{15}H_{21}ON_3$ MW, 259

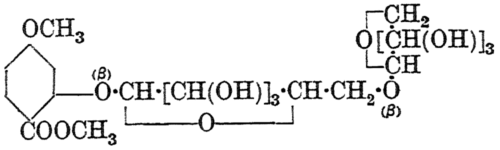
B.p. 175–7°/0·02 mm., 165–70°/0·002 mm. Antimalarial drug.

$B_2,H_2C_2O_4$: m.p. 197–8°.

Elderfield *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 1525.
Wiselogle, *Survey of Antimalarial Drugs*, Vol. 2, 1173.

Primetin.
See 5 : 8-Dihydroxyflavone.

Primeverin



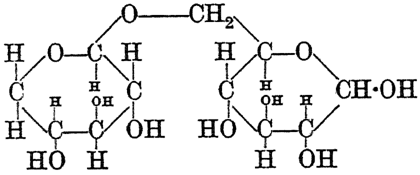
$C_{20}H_{28}O_{13}$ MW, 476

A glycoside from the roots of *Primula officinalis*. Needles from MeOH or EtOH. M.p. 205°. $[\alpha]_D^{20}$ − 76·75° in H_2O. Hyd. ⟶ 4-methoxysalicylic acid + xylose + glucose.

Hexa-acetyl : cryst. from EtOH. M.p. 210–11°.

Jones, Robertson, *J. Chem. Soc.*, 1933, 1618.
Mauthner, *J. prakt. Chem.*, 1941, **159**, 36.

Primeverose (*Glucose*-6-β-d-*xyloside*)



$C_{11}H_{20}O_{10}$ MW, 312

Obtained from various glycosides *e.g.* primeverin and primulaverin from *Primula officinalis*, genticaulin from *Gentiana lutea*, rhamnicosin from *Rhamnus catharticus*. Cryst. M.p. 210° (208°). Sweet taste. Sol. H_2O. Spar. sol. EtOH. $[\alpha]_D^{20}$ + 23·8° ⟶ − 3·4° in H_2O. Reduces Fehling's. Dil. acids ⟶ xylose + *d*-glucose. Br water ⟶ primeverobionic acid.

Phenylosazone : yellow needles from H_2O. M.p. 220°. Sol. EtOH, Me_2CO. Insol. Et_2O, $CHCl_3$. $[\alpha]_D^{19}$ − 109·7° in Py.

α-*Acetyl* : m.p. 201–3°. $[\alpha]_D^{20}$ + 72·3° in $CHCl_3$.

β-*Hepta-acetyl* : needles from EtOH. M.p. 216°. Sol. $CHCl_3$. Spar. sol. EtOH, Et_2O. Insol. H_2O. $[\alpha]_D^{20}$ − 23·5° in $CHCl_3$.

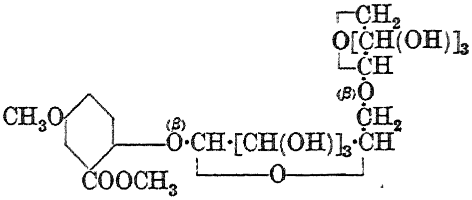
β-*Me-hexa-acetylglycoside* : cryst. from $CHCl_3$–EtOH. M.p. 219–20°. $[\alpha]_D^{20}$ − 37·0° in $CHCl_3$.

α-*Acetochloro* : cryst. from $CHCl_3$–Et_2O. M.p. 201–3°. $[\alpha]_D^{20}$ + 72·1°.

α-*Acetobromo* : colourless needles from Et_2O–pet. ether. M.p. 168–9°.

Helferich, Rauch, *Ann.*, 1927, **455**, 168.
Goris, Vischniac, *Compt. rend.*, 1919, **169**, 871, 975.
Bridel, *Compt. rend.*, 1925, **180**, 1421.
Zemplén, Bognár, *Ber.*, 1939, **72**, 47.

Primulaverin



$C_{20}H_{28}O_{13}$ MW, 476

M.p. 163°. $[\alpha]_D$ − 66·51°. Hyd. ⟶ 5-methoxysalicylic acid + xylose + glucose.

Hexa-acetyl : prisms from MeOH. M.p. 212–14°. $[\alpha]_D^{20}$ −31·2° in $CHCl_3$.

Goris, Vischniac, *Bull. soc. chim.*, 1920, **27**, 262.
Chaudhury, King, Robertson, *J. Chem. Soc.*, 1948, 2220.

Primulin chloride
See Œnin chloride.

Pristane

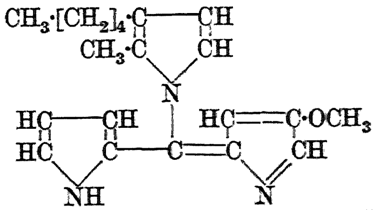
$C_{19}H_{40}$ or $C_{20}H_{42}$ MW, 268 or 282

Obtained from shark liver oil. Oil. Solidifies below 30°. B.p. 296°, 158°/10 mm. D_4^{20} 0·7835. n_D^{15} 1·4410. Sol. C_6H_6, $CHCl_3$, CCl_4, Et_2O, ligroin. Spar. sol. cold Me_2CO, EtOH, warm MeOH.

Toyama, *Chem. Abstracts*, 1923, **17**, 3616.
Sörensen, Mehlum, *Acta Chem. Scand.*, 1948, **2**, 140.
Sörensen, Sörensen, *ibid.*, 166.

Procaine.
See Novocaine.

Prodigiosine



$C_{20}H_{25}ON_3$ MW, 323

Red pigment of *Bacillus prodigiosus*. Brittle solid. Sinters at 70–80°. No definite m.p. Sol. most org. solvents. Insol. H_2O.

B,HClO₄ : needles with metallic lustre from EtOH. Sinters at 226°. M.p. 228°.

Monobenzoyl deriv. : needles from EtOH.Aq. M.p. 170°.

Monosalicyloyl deriv. : needles from EtOH.Aq. M.p. 178°.

Picrate : needles from EtOH. M.p. 176°.

Wrede, Rothhaas, *Z. physiol. Chem.*, 1934, **226**, 95.
Wrede, Hettche, *Ber.*, 1929, **62**, 2678.

Proflavine.
See under 2 : 8-Diaminoacridine.

Profluorane (*Octafluoropropane*)

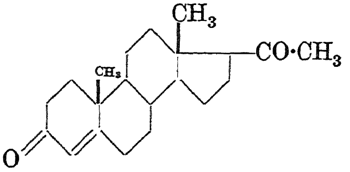
$F_3C{\cdot}CF_2{\cdot}CF_3$

C_3F_8 MW, 188

Gas. F.p. − 183°. B.p. − 36°.

Simons, Block, *J. Am. Chem. Soc.*, 1937, **59**, 1407.

Progesterone (*Progestine*)



$C_{21}H_{30}O_2$ MW, 314

Obtained from *Corpus luteum*. Exists in two forms of the same physiological activity. (α) Prisms from EtOH. M.p. 128·5°. (β) Needles from pet. ether. M.p. 121–2°. $[\alpha]_D$ + 192°.

Readily sol. most org. solvents. Spar. sol. H_2O. $[\alpha]_D$ + 192°. Absorption maximum at 240 mμ. Induces proliferation of uterine mucosa.

Dioxime: cryst. from EtOH.Aq. M.p. 243°.

3-*Enol acetate*: m.p. 135–136·5°.

3-*Enol benzoate*: m.p. 190–2°.

Butenandt, Westphal, Hohlweg, *Z. physiol. Chem.*, 1934, **227**, 84.

Wintersteiner, Allen, *J. Biol. Chem.*, 1934, **107**, 321.

Fieser and Fieser, *Natural Products Related to Phenanthrene, 3rd Ed.*, p. 382. (Reinhold Publishing Corporation, New York.)

Miescher, Kägi, *Helv. Chim. Acta.*, 1939, **22**, 184.

Butenandt, Schmidt-Thome, *Ber.*, 1939, **72**, 182.

Goldberg, Aeschbacher, *Helv. Chim. Acta.*, 1939, **22**, 1185.

Marker, Krueger, *J. Am. Chem. Soc.*, 1940, **62**, 79.

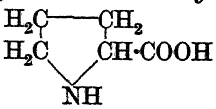
Meystre, Wettstein, Miescher, *Helv. Chim. Acta*, 1947, **30**, 1022.

Progestine.
See Progesterone.

Proguanil.
See Paludrine.

Progynon.
See Œstrone.

Proline (*Pyrrolidine-2-carboxylic acid*)



$C_5H_9O_2N$ MW, 115

d-.

Hygroscopic prisms from EtOH–E t_2O. M.p. 215–20° decomp. $[\alpha]_D^{20}$ + 81·9° in H_2O.

Me ester: $C_6H_{11}O_2N$. MW, 129. $[\alpha]_D^{25}$ + 34°.

N-p-*Toluenesulphonyl*: m.p. 130–3°.

N-m-*Nitrobenzoyl*: prisms from H_2O. M.p. 137–40°. $[\alpha]_D^{20}$ + 120° in *N*-NaOH.

l-.

Cryst. from EtOH–Et_2O. Decomp. at 220–2°. $[\alpha]_D^{20}$ − 80·9° in H_2O.

Me ester: b.p. 70°/10 mm.

Amide: $C_5H_{10}ON_2$. MW, 114. N-*Acetyl*: m.p. 178–80°.

Anhydride: $C_{10}H_{14}O_2N_2$. MW, 194. M.p. 149°. $[\alpha]_D^{19}$ − 147·2°.

N-2 : 4-*Dinitrophenyl*: m.p. 136°.

N-p-*Toluenesulphonyl*: m.p. 130–3°.

Picrate: m.p. 153–4°.

dl-.

Hygroscopic needles from EtOH–Et_2O. M.p. 205° decomp.

Hydrate: $C_{10}H_{18}O_4N_2$, H_2O. M.p. 190–1°. Sol. H_2O, EtOH. Spar. sol. Me_2CO, $CHCl_3$, C_6H_6. Insol. Et_2O.

B,HCl: cryst. from H_2O. M.p. 158–9°.

Et ester: $C_7H_{13}O_2N$. MW, 143. Oil. B.p. 80°/13 mm.

Amide: cryst. from C_6H_6. M.p. 93°. Sol. EtOH, AcOEt, $CHCl_3$.

Anhydride: needles. M.p. 183–4°.

Anilide: cryst. from Me_2CO. M.p. 170°.

N-*Me*: *see* Hygric Acid.

N-m-*Nitrobenzoyl*: prisms from H_2O. M.p. 90–2°.

N-3 : 5-*Dinitrobenzoyl*: prisms from Me_2CO. M.p. 217°.

Picrate: cryst. from EtOH. M.p. 135–7°.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 1122.

Fischer, Zemplén, *Ber.*, 1909, **42**, 2997.

Kapfhammer, Eck, *Z. physiol. Chem.*, 1927, **170**, 294.

Kapfhammer, Matthes, *Z. physiol. Chem.*, 1933, **223**, 43.

Jacobs, Craig, *J. Biol. Chem.*, 1935, **110**, 521.

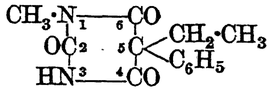
Grassmann, Armin, *Ann.*, 1935, **519**, 192.

Putochin, *Ber.*, 1926, **59**, 1987; 1923, **56**, 2213.

Abderhalden, Sickel, *Z. physiol. Chem.*, 1926, **159**, 163.

Gaudry, Berlinguet, *Chem. Abstracts*, 1949, **43**, 6615.

Prominal (1-*Methyl-5-ethyl-5-phenylbarbituric acid*)

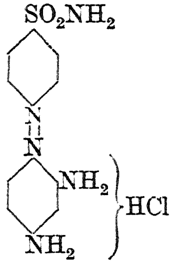


$C_{13}H_{14}O_3N_2$ MW, 246

M.p. 176°.

I.G., F.P., 753,178, (*Chem. Zentr.*, 1934, I, 895).

Prontosil (*4′-Sulphamido-2 : 4-diaminoazobenzene hydrochloride, Streptozon, Rubiazol, Prontosil Red*)



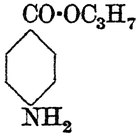
$C_{12}H_{13}O_2N_5$ MW, 259

Cryst. powder. M.p. 247–51°. Sol. 400 parts H_2O. One of a series of substances possessing strong bactericidal properties. Used for treatment of streptococcal and staphylococcal infections, especially erysipelas. Now largely superseded by the more recent " Sulpha " drugs. *See also* Sulphanilamide.

Domagk, *Angew. Chem.*, 1935, **48**, 660.
Goissedet, Despois, Gaillot, Mayer, *Compt. rend. soc. biol.*, 1936, **121**, 1082.

Propadiene.
See Allene.

Propæsin (p-*Aminobenzoic acid propyl ester*)



$C_{10}H_{13}O_2N$ MW, 179

Prisms. M.p. 73–4°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Local anæsthetic.

Fritzsche, D.R.P., 213,459, (*Chem. Zentr.*, 1909, II, 1025).

Propaldehyde.
See Propionaldehyde.

Propanal.
See Propionaldehyde.

Propanalone.
See Pyruvic Aldehyde.

Propandial-1 : 3.
See Glutaraldehyde.

Propane

$CH_3{\cdot}CH_2{\cdot}CH_3$

C_3H_8 MW, 44

Occurs in petroleum gas. F.P. − 189·9°. B.p. − 44·5°, − 124·2°/3 mm. D^0 0·536, $D^{15·9}$ 0·515. Heat of comb. C_p 529·21 Cal. (528·4 Cal.).

Maass, Wright, *J. Am. Chem. Soc.*, 1921, **43**, 1098.
Spencer, Price, *J. Chem. Soc.* 1910, **97**, 388.
Wolkow, Menschutkin, *Chem. Zentr.*, 1900, II, 42.
Timmepmans *Compt. rend.*, 1914, **158**, 790.
Meyer, *Ber.*, 1893, **26**, 2071.

Propane-dicarboxylic Acid.
See Ethylmalonic Acid, Methylsuccinic Acid *and* Glutaric Acid.

Propane 2 : 2-diethyl disulphone.
See Sulphonal.

Propane-1 : 1-disulphonic Acid (*Ethylmethionic acid*)

$CH_3{\cdot}CH_2{\cdot}CH(SO_3H)_2$

$C_3H_8O_6S_2$ MW, 204

Diphenyl ester: $C_{15}H_{16}O_6S_2$. MW, 356. Oil. B.p. 200°/0·005 mm.
Diamide: $C_3H_{10}O_4N_2S_2$. MW, 202. M.p. 169–70°.
Di-trimethylamide: cryst. from Et_2O. M.p. 133°.
Dianilide: cryst. from EtOH. M.p. 151–2°.
Di-[N-Et]-anilide: ethylmethionide. Prisms from EtOH. M.p. 128–9°.
Di-[N-Et]-phenetidide: needles from C_6H_6–pet. ether. M.p. 93·5–94·5°.

Schroeter, *Ann.*, 1918, **418**, 230.
Klaver, *Rec. trav. chim.*, 1935, **54**, 208.

Propane-1 : 2-disulphonic Acid

$CH_3{\cdot}CH{\cdot}SO_3H$
$\quad\quad CH_2{\cdot}SO_3H$

$C_3H_8O_6S_2$ MW, 204

Syrup. Sol. H_2O, EtOH.
Dichloride: $C_3H_6O_4S_2Cl_2$. MW, 241. Plates from pet. ether. M.p. 48°.

Autenrieth, Rudolph, *Ber.*, 1901, **34**. 3477.
Buckton, Hofmann, *Ann.*, 1856, **100**. 153.
Clutterbuck, Cohen, *J. Chem. Soc.*, 1922, **121**, 120.

Propane-1 : 3-disulphonic Acid

$HO_3S{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}SO_3H$

$C_3H_8O_6S_2$ MW, 204

Needles. Decomp. without melting. B.p. 157°/1·4 mm. Sol. H_2O, EtOH.
Dichloride: m.p. 45°.
Diamide: cryst. from H_2O or EtOH. M.p. 169°.
Anhydride: purified by sublimation. M.p. 193–6° decomp.
Dianilide: m.p. 129°.
Di-m-toluidide: pptd. from EtOH with Et_2O. M.p. 222°.
Dihydrazide: needles from EtOH. M.p. 105°.
Di-phenylhydrazide: needles from Me_2CO.Aq. M.p. 177° decomp.

Clutterbuck, Cohen, *J. Chem. Soc.*, 1922, **121**, 120.
Autenrieth, Bernheim, *Ber.*, 1904, **37**, 3808.
Zuffanti, Hendrickson, *J. Am. Chem. Soc.*, 1941, **63**, 2999.
McElwain, Jelinek, Rorig, *J. Am. Chem. Soc.*, 1946, **67** 1580.

Propane-2 : 2-disulphonic Acid (*Dimethylmethionic acid*)

$$\begin{matrix} CH_3 \\ CH_3 \end{matrix} > C < \begin{matrix} SO_3H \\ SO_3H \end{matrix}$$

$C_3H_8O_6S_2$ MW, 204

Diphenyl-ester : m.p. 96°.
Di-[N-Et]-anilide : dimethylmethionide. Cryst. from EtOH. M.p. 130–2°.
Di-[N-Me]-phenetidide : m.p. 114–15°.
Di-[N-Et]-phenetidide : m.p. 109°.

Schroeter, *Ann.*, 1919, **418**, 233.
Klaver, *Rec. trav. chim.*, 1935, **54**, 208.

Propane-dithiol.
See Propylene Dithioglycol *and* Trimethylene Dithioglycol.

Propane-1-sulphonic Acid

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}SO_3H$

$C_3H_8O_3S$ MW, 124

Chloride : $C_3H_7O_2SCl$. MW, 142·5. B.p. 180° decomp., 77·3–78°/13 mm. D_4^{15} 1·2826.
Amide : $C_3H_9O_2NS$. MW, 123. Prisms from Et_2O. M.p. 52°. Sol. H_2O, EtOH.
p-*Toluidide* : cryst. from 95% EtOH. M.p. 67–8°.
Phenylhydrazide : pptd. from EtOH by Et_2O. M.p. 204–5° decomp.

Duguet, *Rec. trav. chim.*, 1902, **21**, 77.
Wagner, Reid, *J. Am. Chem. Soc.*, 1931, **53**, 3411.
Latimer, Bost, *J. Org. Chem.*, 1940, **5**, 25.

Propane-2-sulphonic Acid

$(CH_3)_2CH{\cdot}SO_3H$

$C_3H_8O_3S$ MW, 124

M.p. — 37°. B.p. 159°/1·4 mm. D_4^{25} 1·1877. n_D^{20} 1·4332.
m-*Toluidine salt* : m.p. 109°.
Chloride : oil. B.p. 79°/18 mm.
Amide : cryst. from Et_2O–pet. ether. M.p. 60°. Sol. EtOH, Et_2O. Insol. C_6H_6.

Wagner, Reid, *J. Am. Chem. Soc.*, 1931, **53**, 3411.
Claus, *Ber.*, 1872, **5**, 660; 1875, **8**, 532.
Duguet, *Rec. trav. chim.*, 1906, **25**, 215.
Zuffanti, *J. Am. Chem. Soc.*, 1940, **62**, 1044.

Propane-1 : 2 : 3-tricarboxylic Acid.
See Tricarballylic Acid.

Propanol.
See Propyl Alcohol *and* Isopropyl Alcohol.

Propanolamine.
See Aminopropyl Alcohol.

Propargyl Alcohol (3-*Hydroxyallylene, hydroxymethylacetylene, acetylenylcarbinol*)

$CH{:}C{\cdot}CH_2OH$

C_3H_4O MW, 56

M.p. — 51·80°. B.p. 113·6°/760 mm., 54°/57 mm. D_4^{20} 0·9478. n_D^{20} 1·4320.
Monohydrate : f.p. — 20 to — 60°. D_4^{20} 0·9678. n_D^{20} 1·4120.
Me ether : *see* Methyl propargyl Ether.
Et ether : C_5H_8O. MW, 84. B.p. 82°. D_4^{25} 0·8324. n_D^{20} 1·40390.
Isoamyl ether : $C_8H_{14}O$. MW, 126. B.p. 140°. $D^{12\cdot8}$ 0·84.
Phenyl ether : C_9H_8O. MW, 132. Liq. Decomp. at 210°. D^6 1·246. Insol. H_2O.
Acetyl : b.p. 124–5°. D_4^{20} 1·0052. n_D^{20} 1·42047.
Phenylurethane : m.p. 63°.

Lespieau, *Ann. chim. phys.*, 1912, **27**, 158.
Hatch, Moore, *J. Am. Chem. Soc.*, 1944, **66**, 285.
Henne, Greenlee, *J. Am. Chem. Soc.*, 1945, **67**, 484.
Copenhaver, Bigelow, *Acetylene and Carbon Monoxide Chemistry*, (Reinhold Publishing Corporation, New York), 105.
Eglinton, Jones, Whiting, *J. Chem. Soc.*, 1952, 2873.

Propargylamine (3-*Aminoallylene, 3-aminopropyne*)

$CH{:}C{\cdot}CH_2NH_2$

C_3H_5N MW, 55

B,HCl : unstable. Sol. H_2O, EtOH.
B,HBr : unstable. M.p. 171°.
B,HI : unstable. M.p. 205°.
B,(COOH)₂ : m.p. 143°. Sol. H_2O. Insol. EtOH.
N-*Me* : C_4H_7N. MW, 69. *B,HI* : m.p. 83°. *Oxalate* : m.p. 141°.
N-*Di-Et* : b.p. 120°.
Picrate : plates. M.p. 189°.

Paal, Hermann, *Ber.*, 1889, **22**, 3080.
Paal, Heupel, *Ber.*, 1891, **24**, 3040.

Propargyl bromide (3-*Bromoallylene, 3-bromopropyne*)

$CH{:}C{\cdot}CH_2Br$

C_3H_3Br MW, 119

B.p. 88–90° (82°), 35°/130 mm. D^{19} 1·579. n_D^{19} 1·4942.

Henry, *Ber.*, 1874, **7**, 761.
Kinmann, *Bull. soc. chim.*, 1926, **39**, 698.
v. Braun, Kühn, Siddiqui, *Ber.*, 1926, **59**, 1086.

Propargyl chloride (3-*Chloroallylene, 3-chloropropyne*)

$CH{:}C{\cdot}CH_2Cl$

C_3H_3Cl MW, 74·5

Liq. B.p. 65°. D^5 1·0454.

Henry, *Ber.*, 1875, **8**, 398.

Propargylic Acid.
See Propiolic Acid.

Propargylic Aldehyde.
See Propiolic Aldehyde.

Propene.
See Propylene.

Propenylacetic Acid.
See 2-Ethylidenepropionic Acid.

Propenylacetylene (4-*Methylvinylacetylene*, 3-*penten*-1-*yne*)

$CH_3{\cdot}CH{:}CH{\cdot}C{:}CH$

C_5H_6 MW, 66

Cis: b.p. 44·5°. n_D^{20} 1·4330. *Trans*: b.p. 52·0°. n_D^{20} 1·4368.

Allan, Whiting, *J. Chem. Soc.*, 1953, in the press.

2-Propenylacrylic Acid.
See Sorbic Acid.

3-Propenylallyl Alcohol.
See 2 : 4-Hexadienol-1.

Propenylallylcarbinol (1 : 5-*Heptadienol*-4, 4-*hydroxy*-1 : 5-*heptadiene*)

$CH_3{\cdot}CH{:}CH{\cdot}CH(OH){\cdot}CH_2{\cdot}CH{:}CH_2$

$C_7H_{12}O$ MW, 112

B.p. 156–7° decomp./735 mm., 68–9°/24 mm., 54–5°/6 mm. D_4^{20} 0·8612. $n_D^{13\cdot7}$ 1·45527. Heat with $KHSO_4 \longrightarrow$ 1 : 3 : 5-heptatriene. Oxidised in air.

Auwers, *J. prakt. Chem.*, 1923, **105**, 373.
Enklaar, *Ber.*, 1916, **49**, 211.
I.G., D.R.P., 544,388, (*Chem. Zentr.*, 1932, II, 3156).
Henze, Allen, Leslie, *J. Org. Chem.*, 1942, **7**, 331.

Propenylanisole.
See Anethole *and under* Propenylphenol.

Propenylbenzene.
See β-Methylstyrene.

Propenyl bromide (1-*Bromopropylene*)

$CH_3{\cdot}CH{:}CHBr$

C_3H_5Br MW, 121

B.p. 58–60°/747 mm. D_4^{20} 1·4133. n_D^{20} 1·45193. Insol. H_2O. Alc. KOH $\longrightarrow$ allylene.

Juvala, *Ber.*, 1930, **63**, 1994.
Bachman, *J. Am. Chem. Soc.*, 1933, **55**, 4282.
Schmidt, Kleine, *Ann.*, 1904, **337**, 86.

1-Propenyl-1-butylene.
See 2 : 4-Heptadiene.

Propenylbutyric Acid.
See 4-Heptenic Acid *and* 2-Methyl-3-hexenic Acid.

Propenylcarbinol.
See Crotyl Alcohol.

Propenyl chloride (1-*Chloropropylene*)

$CH_3{\cdot}CH{:}CHCl$

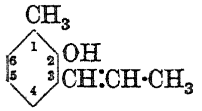
C_3H_5Cl MW, 76·5

Cis:
F.p. — 134·8°. B.p. 32·8°.

Trans:
F.p. — 99°. B.p. 37·4°.

Wislicenus, *Ann.*, 1888, **248**, 297.
Timmermans, *Bull. soc. chim. Belg.*, 1927, **36**, 502.
Reboul, *Ann. chim. phys.*, 1878, **14**, 462.

3-Propenyl-*o*-cresol (1-[2-*Hydroxy*-m-*tolyl*]-*propylene*)



$C_{10}H_{12}O$ MW, 148

Needles from pet. ether. M.p. 41–2°. B.p. 113–15°/12 mm. Sol. most org. solvents, NaOH.

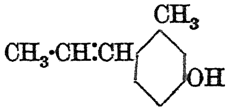
Auwers, Wittig, *Ber.*, 1924, **57**, 1273.

5-Propenyl-*o*-cresol (1-[4-*Hydroxy*-m-*tolyl*]-*propylene*).

Me ether: $C_{11}H_{14}O$. MW, 162. B.p. 121–3°/14 mm. D_4^{16} 0·9844. n_D^{17} 1·5570. *Nitrosochloride*: m.p. 117°.

Klages, *Ber.*, 1904, **37**, 3992.

6-Propenyl-*m*-cresol (1-[4-*Hydroxy*-o-*tolyl*]-*propylene*)

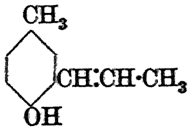


$C_{10}H_{12}O$ MW, 148

Me ether: $C_{11}H_{14}O$. MW, 162. Oil. B.p. 119–21°/13 mm. D_4^{15} 0·9849. n_D^{15} 1·5555. *Nitrosochloride*: needles. M.p. 108°. Spar. sol. Et_2O.

Klages, *Ber.*, 1904, **37**, 3994.

3-Propenyl-*p*-cresol (1-[6-*Hydroxy*-m-*tolyl*]-*propylene*)



$C_{10}H_{12}O$ MW, 148

B.p. 126–8°/14 mm., 120–4°/11 mm. D_4^{20} 1·019. n_D^{20} 1·5727.
Me ether: $C_{11}H_{14}O$. MW, 162. B.p. 127·5–128°/19 mm. D_4^{20} 0·979. n_D^{20} 1·5522.

Auwers, *Ann.*, 1917, **413**, 299.

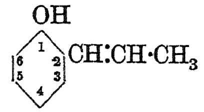
2-Propenylcrotonic Acid.
See 2-Methylsorbic Acid.

Propenylethylidenepropane.
See 2 : 6-Octadiene.

Propenylethynylcarbinol.
See 4-Hexen-1-yn-3-ol.

Propenylguaiacol.
See Isochavibetol *and* Isoeugenol.

o-Propenylphenol (o-*Hydroxy*-β-*methylstyrene*)



$C_9H_{10}O$ MW, 134

Needles from ligroin. M.p. 37–8°. B.p. 230–1°, 112–13°/12 mm. $D_4^{13\cdot9}$ 1·0441. $n_D^{14\cdot2}$ 1·584.

Me ether: o-propenylanisole. $C_{10}H_{12}O$. MW, 148. Volatile oil. B.p. 223–4°/751 mm., 104–5°/13 mm. D_4^{15} 0·9962. n_D^{15} 1·560.

Et ether: o-propenylphenetole. $C_{11}H_{14}O$. MW, 162. Oil. B.p. 230–1°/757 mm., 120–1°/17 mm. D_4^{24} 0·97307. n_D^{24} 1·544.

Claisen, *Ann.*, 1919, **418**, 86.
Pauly, v. Buttlar, *Ann.*, 1911, **383**, 280.
Klages, *Ber.*, 1904, **37**, 3987.

m-Propenylphenol (m-*Hydroxy*-β-*methylstyrene*).

Me ether: m-propenylanisole. Liq. B.p. 226–9°. D^0 1·0013.

Et ether: m-propenylphenetole. Volatile oil. B.p. 124–6°/16 mm. D_4^{22} 0·9782. n_D^{22} 1·542.

Nitrosochloride: m.p. 122–3°.

Moureu, *Bull. soc. chim.*, 1896, **15**, 1024.
Klages, *Ber.*, 1904, **37**, 3989.

p-Propenylphenol.
See Anol.

Propenylphenylcarbinol (1-*Phenyl*-2-*butenol*-1, α-*propenylbenzyl alcohol*, 1-*phenylcrotyl alcohol*)

$CH_3 \cdot CH{:}CH \cdot CH(OH) \cdot C_6H_5$

$C_{10}H_{12}O$ MW, 148

B.p. 121·5–123·5°/14 mm., 88–90°/1 mm. n_D^{20} 1·5525.

Acetyl: b.p. 135–6°/21·5 mm. n_D^{25} 1·5087.

Succinate: m.p. 72–80°.

Acid succinate: m.p. 76–8°.

Acid phthalate: small needles from Et_2O–pet. ether. M.p. 93–4°.

p-*Nitrobenzoyl*: prismatic needles from Et_2O–pet. ether. M.p. 99°.

p-*Xenylurethane*: prismatic needles from pet. ether. M.p. 124°.

d-.

Acid phthalate: glistening needles. M.p. 80–1°. $[\alpha]_D$ + 15·5° in Et_2O.

l-.

B.p. 126°/20 mm. n_D^{20} 1·5525. $[\alpha]_{5790}^{20}$ — 29·16°.

Acid phthalate: long glistening needles from Et_2O-pet. ether. M.p. 81–2°. $[\alpha]_D$ — 17·1° in Et_2O.

p-*Nitrobenzoyl*: oil. $[\alpha]$ — $_{5790}$46·3° in Py.

p-*Xenylurethane*: hairy needles from methylene chloride-pet. ether. M.p. 120°. $[\alpha]_D$ + 20·0°.

Ingold, Wilson, *J. Chem. Soc.*, 1933, 1497.
Burton, *J. Chem. Soc.*, 1929, 457.
Kenyon, Partridge, Phillips, *J. Chem. Soc.*, 1937, 207.

Propenyl phenyl Ketone.
See Crotonophenone.

2-Propenylpiperidine.
See β-Coniceine.

2-Propenylpropylene.
See 2-Methyl-1 : 3-pentadiene.

2-Propenylpyridine

C_8H_9N MW, 119

Liq. B.p. 189–90°, 70–74°/15 mm. D^0 0·9595. Spar. sol. H_2O. Volatile in steam.

B,HAuCl$_4$: needles from HCl.Aq. M.p. 135–6°.

B$_2$,*H*$_2$*PtCl*$_6$: needles. M.p. 185–6° decomp.

Picrate: yellow needles from Et_2O. M.p. 165–6°.

Ladenburg, *Ann.*, 1888, **247**, 26.

Note.—This compound is referred to in the above reference as α-allylpyridine.

4-Propenylpyridine.
Liq. B.p. 200–2°. Volatile in steam.

B,HCl: cryst. M.p. 247°.

B,HAuCl$_4$: yellow needles from HCl.Aq. M.p. 174° decomp.

B,HCl,HgCl$_2$: needles from H_2O. M.p. 150°.

B$_2$,*H*$_2$*PtCl*$_6$: needles. M.p. 206° decomp.

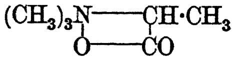
Picrate: yellow needles from H_2O. M.p. 169–70° decomp.

Ahrens, *Ber.*, 1905, **38**, 157.

4-Propenylveratrol.
See under Isoeugenol.

p-Propenyltoluene.
See 1-p-Tolylpropylene.

α-Propiobetaine (*Homobetaine, alaninebetaine, methylbetaine*)



$C_6H_{13}O_2N$ MW, 131

l-.

Plates from EtOH–Et_2O. Decomp. at 242°. $[\alpha]_D^{20}$ — 19·7° in H_2O.

B,HAuCl$_4$: dimorphous. (i) Yellow plates from dil. HC. M.p. 259° decomp. (ii) Cryst. from H_2O. M.p. 226° decomp.

dl-.
Cryst.
B,HAuCl₄: needles from 1% HCl. M.p. 240°.
B_2,H_2PtCl_6: orange plates + $2H_2O$ from H_2O. M.p. 210–12° decomp.

Fischer, *Ber.*, 1907, **40**, 5002.
Kossel, Edlbacher, *Z. physiol. Chem.*, 1919, **107**, 45.

Propiodinitrile.
See Dipropionitrile.

Propioin.
See Diethylketol.

β-Propiolactam (2-*Azetidinone*)

$$\begin{array}{l} H_2C—CO \\ H_2C—NH \end{array}$$

C_3H_5ON MW, 71

Colourless cryst. M.p. 73–4°. B.p. 106°/15 mm. Sol. EtOH, Et_2O, $CHCl_3$.

Holley, Holley, *J. Am. Chem. Soc.*, 1949, **71**, 2129.

Propiolic Acid (*Acetylene-carboxylic acid, propargylic acid*)

CH:C·COOH

$C_3H_2O_2$ MW, 70

Liq. with odour resembling acetic acid. M.p. 18°; monohydrate cryst. felted mass, m.p. 0·3°; $3C_3H_2O_2 + 1H_2O$, needles, m.p. 10°. B.p. 144° decomp., 102°/200 mm., 83–4°/50 mm. Misc. with H_2O, EtOH, Et_2O, $CHCl_3$. D_4^{20} (anhyd.) 1·1380. n_α^{20} 1·43064. Red. ⟶ propionic acid. HCl, HBr, HI ⟶ β-halogen-acrylic acid. Reduces $AgNO_3$ and $HgCl_2$ and platinic chloride sols. in warm. Forms explosive Ag and Cu comps. Cu_2Cl_2 ⟶ cryst. add. comp.
K salt: cryst. + $1H_2O$. Decomp. at 105°. Very sol. H_2O.
Aniline salt: prisms + $\frac{1}{2}H_2O$ from EtOH.Aq. M.p. 71–2°.
Me ester: $C_4H_4O_2$. MW, 84. B.p. 102°/742 mm. Lachrymatory.
Et ester: $C_5H_6O_2$. MW, 98. B.p. 119°/745 mm. D_{25}^{25} 0·9583. n_α^{15} 1·4133. Red. ⟶ ethyl propargyl ether. $NH_2{\cdot}NH_2$ ⟶ pyrazolone.
Anhydride: $C_6H_2O_3$. MW, 122. B.p. 56°/16 mm. D_4^{21} 1·1432. $n_D^{23\cdot4}$ 1·4358. Spar. sol. ligroin.
Nitrile: C_3HN. MW, 51. M.p. 5°. B.p. 42·5°. D_4^{17} 0·8159. n_D^{17} 1·38699. Spar. sol. H_2O. Sols. rapidly decompose. Decomp. by air and light. KOH ⟶ resin. $AgNO_3$ ⟶ white ppt. Vapour affects mucous membrane.
Amide: C_3H_3ON. MW, 69. Needles or plates from Et_2O. M.p. 61–2°. Very sol. H_2O, EtOH, Et_2O. Spar. sol. $CHCl_3$, cold C_6H_6, CS_2.
Anilide: needles from H_2O. M.p. 86–7°.

Owen, Sultanbawa, *J. Chem. Soc.*, 1949, 3111.
Backer, Beute, *Rec. trav. chim.*, 1935, **54**, 168.
Chéou, *Chem. Abstracts*, 1935, 2513.
Straus, Heyn, Schwener, *Ber.*, 1930, **63**, 1090.
Straus, Voss, *Ber.*, 1926, **59**, 1685.
Moureu, André, *Compt. rend.*, 1913, **157**, 897.
Perkin, Simonsen, *J. Chem. Soc.*, 1907, **91**, 834.
Baeyer, *Ber.*, 1885, **18**, 677.

Propiolic Aldehyde (*Propargylic aldehyde*)

CH:C·CHO

C_3H_2O MW, 54

Thin oil. B.p. 59–61°. Misc. with H_2O. Affects eyes and mucous membranes. $NH_3.AgNO_3$ ⟶ white ppt. $NH_3{\cdot}CuCl_2$ ⟶ yellowish-red ppt. Dil. alkalis ⟶ acetylene + formic acid. NH_2OH ⟶ isoxazole.
Di-Me acetal: $C_5H_8O_2$. MW, 100. B.p. 110°.
Di-Et acetal: $C_7H_{12}O_2$. MW, 128. B.p. 139–41°. NH_2OH ⟶ isoxazole. $NH_2{\cdot}NH_2$ ⟶ pyrazole. *Cu deriv.*: m.p. 160°.
p-*Nitrophenylhydrazone*: m.p. 123–4°.

Grard, *Ann. chim.*, 1930, **13**, 336.
Claisen, *Ber.*, 1903, **36**, 3667; 1898, **31**, 1022.

Propionaldehyde (*Propanal, propaldehyde, propionic aldehyde*)

$CH_3{\cdot}CH_2{\cdot}CHO$

C_3H_6O MW, 58

Liq. with suffocating odour. F.p. −81°. B.p. 47·5–49°. Sol. 5 parts H_2O at 20°. D_{25}^{25} 0·79664. n_D^{20} 1·3636. Heat of comb. C_p 434·35 Cal., C_v 433·8 Cal. HCl ⟶ metapropionaldehyde and parapropionaldehyde. KOH ⟶ propionaldol. Red. ⟶ propyl alcohol. *m*-Dinitrobenzene + KOH ⟶ greenish-brown col. Sodium nitroprusside and piperidine ⟶ blue col. (green to blue in dil. sol.). Forms bisulphite comp.
Metapropionaldehyde: $(C_3H_6O)_n$. Cryst. M.p. 180°. Insol. H_2O. Spar. sol. EtOH. Sublimes. Dist. with little HCl ⟶ propionaldehyde.
Parapropionaldehyde: $C_9H_{18}O_3$. MW, 174. F.p. −20° to needles. B.p. 172–3°/773 mm., 85–6°/50 mm. Misc. with EtOH, Et_2O. Spar. sol. H_2O. D_{16}^{16} 0·9445. Dist. with little HCl or H_2SO_4 ⟶ propionaldehyde.
Propionaldehyde-ammonia: C_3H_9ON. MW, 75. Flocculent ppt. Mod. sol. EtOH, Et_2O. Spar. sol. ligroin. Gradually decomp. at room temp.
Bisulphite comp.: cryst. + $1H_2O$. Spar. sol. EtOH.

Oxime: C_3H_7ON. MW, 73. M.p. 40°. B.p. 130–2°, 77°/100 mm. D_4^{20} 0·9258. n_D^{20} 1·4287.

Semicarbazone: two forms. (i) Needles from C_6H_6–ligroin. M.p. 88–90°. Very easily sol. H_2O, hot C_6H_6. (ii) Plates from H_2O. M.p. 154°. Mod. sol. C_6H_6.

Phenylhydrazone: oil. B.p. 205°/108 mm. Becomes red in air. *Picrate*: pale yellow needles from EtOH. M.p. 156–7°.

Diphenylhydrazone: needles from EtOH.Aq. M.p. 20–1°.

Methylphenylhydrazone: yellow oil. B.p. 198°/170 mm.

o-*Nitrophenylhydrazone*: orange needles from EtOH.Aq. M.p. 72°.

m-*Nitrophenylhydrazone*: orange-yellow plates from EtOH.Aq. M.p. 83°.

p-*Nitrophenylhydrazone*: orange-yellow needles from EtOH.Aq. M.p. 125°.

p-*Iodophenylhydrazone*: m.p. 196°.

o-*Tolylsemicarbazone*: plates. M.p. 129–30°.

p-*Tolylsemicarbazone*: plates. M.p. 135–7°.

Benzoylhydrazone: prisms from hot H_2O. M.p. 117°. Very sol. EtOH, Et_2O, $CHCl_3$. Easily decomp. by acids and alkalis.

Cyanhydrin: *see under* 1-Hydroxybutyric Acid.

Di-Me acetal: $C_5H_{12}O_2$. MW, 104. B.p. 89°. D^{21} 0·849. n^{21} 1·3799.

Di-Et acetal: $C_7H_{16}O_2$. MW, 132. B.p. 123°. D^0 0·8825, D_4^{25} 0·833. $n_D^{25 \cdot 5}$ 1·3872.

Di-propyl acetal: b.p. 157–60°/760 mm. D_4^{22} 0·8223. n_D^{24} 1·4038.

Anil: propylideneaniline. Dimer. $C_{18}H_{22}N_2$. Needles from EtOH. M.p. 103–4°. Sol. Et_2O, C_6H_6. Spar. sol. cold EtOH.

Sah, Shih, *J. Chinese Chem. Soc.*, 1935, **3**, 246.

Bauer, Strauss, *Ber.*, 1932, **65**, 311.

Hurd, Meinert, *Organic Syntheses*, 1932, XII, 64.

I.G., B.P., 354,388, (*Chem. Zentr.*, 1931, II, 2932).

Kirrmann, *Ann. chim.*, 1929, **11**, 223.

Wood, Comley, *J. Soc. Chem. Ind.*, 1923, **42**, 429T.

Harries, Oppenheim, *Chem. Zentr.*, 1916, II, 992.

Nef, *Ann.*, 1904, **335**, 202.

Propionamide

$CH_3 \cdot CH_2 \cdot CONH_2$

C_3H_7ON MW, 73

Plates from C_6H_6. M.p. 81·3°. B.p. 213°. Sol. H_2O, EtOH, Et_2O, $CHCl_3$. Volatile in steam. Heat of comb. C_p 439·8 Cal. Combines with min. acids, Na and K. Na $\longrightarrow$ sodium dipropionamide. Red. $\longrightarrow$ propylamine and dipropylamine.

Hg comp.: plates. M.p. 202°. Spar. sol. cold H_2O.

N-*Formyl*: needles from C_6H_6. M.p. 65°. Sol. H_2O, EtOH, Et_2O.

N-*Acetyl*: m.p. 86° (81–2°). B.p. 230–40°.

N-*Benzoyl*: needles or prisms. M.p. 98°.

N-*Di-Me*: $C_5H_{11}ON$. MW, 101. B.p. 165–78°.

N-*Di-Et*: $C_7H_{15}ON$. MW, 129. B.p. 191°. Sol. H_2O, acids.

N-*Chloro*: cryst. from C_6H_6. M.p. 34°.

N-*Bromo*: needles. M.p. 80°. Sol. H_2O, EtOH, Et_2O, Me_2CO, $CHCl_3$. Spar. sol. cold C_6H_6, pet. ether.

N-*Iodo*: m.p. 128° decomp. Sol. EtOH, Me_2CO. Mod. sol. AcOH. Spar. sol. C_6H_6, pet. ether.

Mitchell, Reid, *J. Am. Chem. Soc.*, 1931, **53**, 1881.

Soc. Française de Catalyse Généralisée, F.P., 701,579, (*Chem. Zentr.*, 1931, II, 1193).

Hoffmann, *Ber.*, 1882, **15**, 981.

Propionaphthone.

See Ethyl naphthyl Ketone.

Propione.

See Diethyl Ketone.

Propionic Acid

$CH_3 \cdot CH_2 \cdot COOH$

$C_3H_6O_2$ MW, 74

Colourless liq. with acrid odour. F.p. −19·7°. B.p. 141·35°. D_{15}^{15} 0·99874. n_D^{22} 1·3859. Misc. in all proportions with H_2O. Heat of comb. 367·4 Cal. Mol. b.p. elevation 35·1. $k = 1{\cdot}22 \times 10^{-5}$ at 25°. Most salts sol. H_2O.

NH_4 salt: very hygroscopic cryst. M.p. 45°. Sol. H_2O, EtOH, AcOH.

K salt: plates $+ 1H_2O$ from EtOH.Aq. Becomes anhyd. at 120° and does not melt below 300°.

Benzylamine salt: cryst. from CCl_4. M.p. 68° (46·5–47°). Very sol. H_2O, EtOH. Spar. sol. C_6H_6. Insol. pet. ether.

Me ester: *see* Methyl propionate.

Et ester: $C_5H_{10}O_2$. MW, 102. F.p. − 72·6°. B.p. 99·1°. D_4^{25} 0·8830. n_D^{20} 1·38385.

Propyl ester: $C_6H_{12}O_2$. MW, 116. B.p. 122°. D^{20} 0·8809.

Isopropyl ester: b.p. 109–11°/750 mm. D^0 0·8931.

Butyl ester: *see* Butyl propionate.

sec.-*Butyl ester*: *see sec.*-Butyl propionate.

Isobutyl ester: *see* Isobutyl propionate.

n-*Amyl ester*: $C_8H_{16}O_2$. MW, 144. F.p. − 73·1°. B.p. 168·65°. D_4^{15} 0·8761. n_D^{15} 1·4096.

Isoamyl ester: *see* Isoamyl propionate.

tert.-*Amyl ester*: *see tert.*-Amyl propionate.

Decyl ester: $C_{13}H_{26}O_2$. MW, 214. B.p. 124°/8 mm. D_4^{20} 0·8639. n_D^{20} 1·42907.

Glycerol mono-ester: *see* Propionin.

Glycerol di-ester: dipropin. $C_9H_{16}O_5$. MW, 204. B.p. 170–3°/10 mm.

Glycerol tri-ester: tripropin. $C_{12}H_{20}O_6$. MW, 260. B.p. 177–82°/20 mm., 130–2°/3 mm. D^{20}_{18} 1·100. n^{19}_D 1·43175.

Phenyl ester: $C_9H_{10}O_2$. MW, 150. Prisms. M.p. 20°. B.p. 211°.

Benzyl ester: $C_{10}H_{12}O_2$. MW, 164. B.p. 222°.

Chloride: propionyl chloride. C_3H_5OCl. MW, 92·5. B.p. 80°. D^{20}_4 1·0646. n^{20}_D 1·40507.

Bromide: propionyl bromide. C_3H_5OBr. MW, 137. B.p. 103–103·6°/770 mm. $D^{16·4}$ 1·5210. $n^{16·4}_D$ 1·45783.

Amide: *see* Propionamide.

Anhydride: $C_6H_{10}O_3$. MW, 130. B.p. 168·4–168·8°/780 mm., 67·5°/18 mm. D^{15} 1·0169.

Nitrile: propionitrile, ethyl cyanide. C_3H_5N. MW, 55. Liq. with ethereal odour. F.p. – 103·5°. B.p. 97°. D^{25}_4 0·7770. n^{25}_D 1·3659. Part misc. with H_2O. Mol. b.p. elevation 22·6. Heat of comb. C_v 446·7 Cal. Na, K $\longrightarrow$ di- and tri-molecular comps. AcOH $\longrightarrow$ *N*-acetylpropionamide. Extremely poisonous.

Hydrazide: cryst. M.p. 40°. B.p. 130°/16 mm. Sol. H_2O. Spar. sol. Et_2O.

Anilide: propionanilide. Plates from EtOH.Aq., Et_2O, or C_6H_6. M.p. 105–6°. Mod. sol. hot H_2O, EtOH, Et_2O. Spar. sol. cold H_2O. Heat of comb. C_p 1168·0 Cal.

o-*Nitroanilide*: lemon-yellow cryst. from EtOH. M.p. 63°. Sol. boiling H_2O, EtOH, Et_2O, C_6H_6.

p-*Nitroanilide*: yellowish-brown plates from Ac_2O. M.p. 182°.

o-*Toluidide*: cryst. M.p. 18°. B.p. 144–5°/11 mm.

m-*Toluidide*: needles from Et_2O. M.p. 81°. Sol. EtOH, Et_2O, ligroin.

p-*Toluidide*: plates from C_6H_6. M.p. 126°. Sol. EtOH, Et_2O, Me_2CO, AcOH, $CHCl_3$, C_6H_6. Spar. sol. cold H_2O, ligroin.

1-*Naphthylamide*: cryst. from EtOH. M.p. 116°.

Hardy, *J. Chem. Soc.*, 1936, 361, 365.
du Pont, U.S.P., 2,015,065, (*Chem. Zentr.*, 1936, I, 1310); U.S.P., 1,979,717, (*Chem. Abstracts*, 1935, **29**, 181).
I.G., U.S.P., 2,005,183, (*Chem. Abstracts*, 1935, **29**, 5125).
Werkmann, Rayburn, Hixon, U.S.P., 1,991,993, (*Chem. Abstracts*, 1935, **29**, 2298).
Timmermans, Hennaut-Roland, *J. chim. phys.*, 1930, **27**, 401.
Pierre, Puchot, *Ann. chim.*, 1873, **28**, 75.
Auger, *Compt. rend.*, 1907, **145**, 1289.
Hanriot, Bouveault, *Bull. soc. chim.*, 1889, **1**, 171.
Simons, *J. Am. Chem. Soc.*, 1926, **48**, 1993.

Propionin (*Monopropin, glycerol α-propionate*)

$CH_2·O·CO·CH_2·CH_3$
$CHOH$
CH_2OH

$C_6H_{12}O_4$ MW, 148

B.p. 132–4°/3 mm. D^{20}_4 1·1537.

Diacetyl: $C_{10}H_{16}O_6$. MW, 232. B.p. 150°/12 mm.

Eastman Kodak, U.S.P., 2,005,371, (*Chem. Zentr.*, 1935, II, 2446).
Schuette, Hale, *J. Am. Chem. Soc.*, 1930, 52, 1979.
Abderhalden, Eichwald, *Ber.*, 1914, **47**, 1859.

Propionitrile.
See under Propionic Acid.

Propionoin.
See Diethylketol.

Propionylacetic Acid (2-*Keto*-n-*valeric acid*)

$CH_3·CH_2·CO·CH_2·COOH$

C_5H_8O MW, 116

Me ester: $C_6H_{10}O_3$. MW, 130. B.p. 73–6°/16 mm.

Et ester: $C_7H_{12}O_3$. MW, 144. B.p. 191°, 75–8°/9 mm. Alc. $FeCl_3$ $\longrightarrow$ red. col. Forms bisulphite comp.

Nitrile: C_5H_7ON. MW, 97. B.p. 164–5°. D^9 0·976. Insol. H_2O.

Cu deriv.: cryst. from C_6H_6–pet. ether. M.p. 160–1°.

Décombe, *Ann. chim.*, 1932, **18**, 81.
I.C.I., F.P., 666,703, (*Chem. Abstracts*, 1930, **24**, 1120).
Breckport, *Chem. Abstracts*, 1924, **18**, 816.
Wahl, *Compt. rend.*, 1911, **152**, 97.
Dupont, *Compt. rend.*, 1909, **148**, 1524.
Reymenant, *Chem. Zentr.*, 1901, I, 95.
Riegel, Lilienfeld, *J. Am. Chem. Soc.*, 1945, **67**, 1274.

Propionylacetone (*Acetylpropionylmethane, hexandione*-2 : 4, 2 : 4-*diketohexane*)

$CH_3·CH_2·CO·CH_2·CO·CH_3$

$C_6H_{10}O_2$ MW, 114

Oil. B.p. 158°. D^{20}_4 0·959. n^{20}_D 1·4516.

Cu deriv.: blue needles. M.p. 197–8° (179°).

Powell, Seymour, *J. Am. Chem. Soc.*, 1931, **53**, 1049.
Fittig, *Ann.*, 1907, **353**, 24.
Claisen, Ehrhardt, *Ber.*, 1889, **22**, 1014.
Adams, Hauser, *J. Am. Chem. Soc.*, 1945, **67**, 284.

Propionylanisole.
See under 2-, *and* 4-Hydroxypropiophenone.

Propionylbenzene.
See Propiophenone.

2-Propionylbenzoic Acid.
See Propiophenone-*o*-carboxylic Acid.

3-Propionyl-*sec.*-*n*-butyl Alcohol.
See 3-Methyl-2-hexanolone-4.

4-Propionyl-1-butylene.
See 1-Heptenone-5.

2-Propionyl-2-butylene.
See 3-Methyl-2-hexenone-4.

3-Propionylbutyric Acid.
See 4-Keto-*n*-heptylic Acid.

Propionylbutyryl.
See Heptandione-3 : 4.

Propionylbutyrylmethane.
See Octandione-3 : 5.

5-Propionyl-*n*-caproic Acid.
See 6-Ketopelargonic Acid.

Propionylcaproyl (*Nonandione*-3 : 4, *ethyl* n-*amyl diketone*, 3 : 4-*diketononane*)

$CH_3\cdot[CH_2]_4\cdot CO\cdot CO\cdot CH_2\cdot CH_3$

$C_9H_{16}O_2$ MW, 156

B.p. 77–80°/10 mm. D_4^0 0·927.

3-*Oxime*: needles. M.p. 33–4°. B.p. 131–2°/9 mm. Spar. sol. most org. solvents.

Dioxime: needles from boiling C_6H_6. M.p. 158°. Sol. EtOH, Et_2O. Spar. sol. pet. ether. Sublimes.

Locquin, *Bull. soc. chim.*, 1904, **31**, 1176.

7-Propionylcaprylic Acid.
See 8-Ketoundecylic Acid.

Propionylcarbinol (*Ethylketol*, 2-*keto*-n-*butyl alcohol*, *hydroxymethyl ethyl ketone*, 1-*butanolone*-2)

$CH_3\cdot CH_2\cdot CO\cdot CH_2OH$

$C_4H_8O_2$ MW, 88

Colourless liq. B.p. 160°. Misc. with H_2O, EtOH, Et_2O. D_{15}^{15} 1·0365. $n_D^{14\cdot5}$ 1·4315. Reduces Fehling's. Acid $KMnO_4$ ⟶ propionic acid. Forms bisulphite comp.

Formyl: $C_5H_8O_3$. MW, 116. B.p. 176–8°. D_{17}^{17} 1·0946. n_D^{17} 1·424. *Semicarbazone*: cryst. from $CHCl_3$ or C_6H_6. M.p. 115°.

Acetyl: b.p. 79°/14 mm. n_D^{15} 1·4212.

Benzoyl: b.p. 150–2°/8 mm. n_D^{14} 1·5162. *Semicarbazone*: flat needles from EtOH. M.p. 147°.

Me ether: $C_5H_{10}O_2$. MW, 102. B.p. 130–1°/729 mm. *Phenylhydrazone*: yellow oil. B.p. 170°/18 mm.

Et ether: $C_6H_{12}O_2$. MW, 116. Colourless oil turning yellow in air. B.p. 146°, 55°/24 mm. D_4^{16} 0·914. *Semicarbazone*: m.p. 87°.

Isobutyl ether: $C_8H_{16}O_2$. MW, 144. B.p. 68–9°/13 mm. *Semicarbazone*: m.p. 72°.

Oxime: cryst. from $CHCl_3$. M.p. 60–1°.

Semicarbazone: cryst. from EtOH. M.p. 136–8°. Spar. sol. H_2O.

Sommelet, *Bull. soc. chim.*, 1911, **9**, 35.
Kling, *Compt. rend.*, 1905, **140**, 1345.

Propionylcatechol.
See Dihydroxypropiophenone.

Propionylcresol.
See Hydroxymethylpropiophenone.

Propionyl cyanide.
See under 1-Ketobutyric Acid.

Propionylcyclohexane.
See Hexahydropropiophenone.

Propionylethylene.
See Ethyl vinyl Ketone.

Propionylformic Acid.
See 1-Ketobutyric Acid.

2-Propionylheptane.
See 4-Methylnonanone-3.

6-Propionyl-*n*-heptylic Acid.
See 7-Ketocapric Acid.

Propionyl hydroperoxide.
See Perpropionic Acid.

Propionylhydroquinone.
See Dihydroxypropiophenone.

1-Propionylisobutylene.
See 2-Methyl-2-hexenone-4.

Propionylnaphthalene.
See Ethyl naphthyl Ketone.

Propionylnaphthol.
See Ethyl hydroxynaphthyl Ketone.

***p*-Propionylphenetole.**
See under 4-Hydroxypropiophenone.

Propionylphenol.
See 2-, *and* 4-Hydroxypropiophenone.

2-Propionylpiperidine.
See Conhydrinone.

3-Propionylpropanol-1.
See 1-Hexanolone-4.

1-Propionylpropionic Acid (1-*Methylpropionylacetic acid*, 2-*keto*-1-*methyl*-n-*valeric acid*)

$$CH_3\cdot CH_2\cdot CO\cdot \overset{CH_3}{\overset{|}{C}H}\cdot COOH$$

$C_6H_{10}O_3$ MW, 130

Me ester: $C_7H_{12}O_3$. MW, 144. Liq. with aromatic odour. B.p. 187°, 80°/12 mm. n_D^{25} 1·4211.

Et ester: $C_8H_{14}O_3$. MW, 158. Oil. B.p. 199°, 87–8°/16 mm. D^{15} 0·9827. Forms Na deriv. Red. ⟶ 2-hydroxy-1-methyl-*n*-valeric acid. NaOH ⟶ diethyl ketone. $C_6H_5\cdot NH\cdot NH_2$ ⟶ 4-methyl-3-ethyl-1-phenylpyrazolone-5. *Di-Et-acetal*: b.p. 223–6°.

Isobutyl ester: $C_{10}H_{18}O_3$. MW, 186. B.p. 99–100°/11 mm.

Amide: $C_6H_{11}O_2N$. MW, 129. Hygroscopic cryst. M.p. 82°. Sol. H_2O.

Nitrile: C_6H_9ON. MW, 111. Oil with sweet odour. B.p. 193·5°. D^0 0·9728.

Schroeter, *Ber.*, 1916, **49**, 2719.
Pingel, *Ann.*, 1888, **245**, 84.
Hantzsch, Wohlbrück, *Ber.*, 1887, **20**, 1320.
Hauser, Walker, *J. Am. Chem. Soc.*, 1947, **69**, 295.

2-Propionylpropionic Acid.
See 3-Keto-*n*-caproic Acid.

3-Propionyl-*n*-propyl Alcohol.
See 1-Hexanolone-4.

Propionylpropylene.
See 1-Hexenone-4, 2-Hexenone-4 *and* 2 Methyl-1-pentenone-3.

Propionylresorcinol.
See Dihydroxypropiophenone.

β-Propionylstyrene.
See Ethyl styryl Ketone.

Propionylthiophene.
See Propiothienone.

Propionyltoluene.
See Ethyl tolyl Ketone.

4-Propionyl-*n*-valeric Acid.
See 5-Keto-*n*-caprylic Acid.

Propionylvaleryl.
See Octandione-3 : 4.

Propiophenone (*Ethyl phenyl ketone, propionylbenzene*)

$C_6H_5{\cdot}CO{\cdot}CH_2{\cdot}CH_3$

$C_9H_{10}O$ MW, 134

Plates. M.p. 19–20°. B.p. 218°/764 mm., 115–20°/21 mm., 75°/3 mm. D_{25}^{25} 1·0087. n_D^{20} 1·534 (1·5256). CrO_3 ⟶ benzoic and acetic acids. Na in EtOH ⟶ ethylphenylcarbinol. Zn + HCl or H (+ Ni) ⟶ propylbenzene.

Oxime: plates from pet. ether. M.p. 53–4°. B.p. 245–6° (decomp.), 165°/38 mm.

2 : 4-*Dinitrophenylhydrazone*: red leaflets from EtOH. M.p. 187–9°.

Semicarbazone: needles from EtOH. M.p. 182° (173°). Spar. sol. EtOH, Et_2O.

Di-Me acetal: $C_{11}H_{16}O_2$. MW, 180. Oil. B.p. 206–8°, 92–3°/18 mm. D_4^{15} 0·9888.

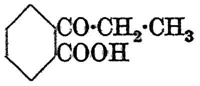
Imide: b.p. 102°/13 mm.

Azine: yellow needles. M.p. 79–80° (68°). Sol. EtOH.

Anil: pale yellow needles from hexane. M.p. 50°. B.p. 169°/11 mm. Spar. sol. pet. ether.

Thompson, Stevens, *J. Chem. Soc.*, 1932, 2611.
Shriner, Turner, *J. Am. Chem. Soc.*, 1930, **52**, 1267.
Straus, Berkov, *Ann.*, 1913, **401**, 140.
Auwers, *Ber.*, 1912, **45**, 996.
Hauser, Humphlett, Weiss, *J. Am. Chem. Soc.*, 1948, **70**, 426.

Propiophenone-*o*-carboxylic Acid (2-*Propionylbenzoic acid*)



$C_{10}H_{10}O_3$ MW, 178

Needles from EtOH.Aq., or C_6H_6. M.p. 93° (97°). NaHg ⟶ 3-ethylphthalide.

Me ester: $C_{11}H_{12}O_3$. MW, 192. B.p. 157–8°/19 mm. $D_4^{16\cdot4}$ 1·1274. $n_D^{16\cdot4}$ 1·5197.

Amide: $C_{10}H_{11}O_2N$. MW, 177. Needles from H_2O. Sinters at 150°. M.p. 159°. Spar. sol. $CHCl_3$, ligroin, C_6H_6, CS_2.

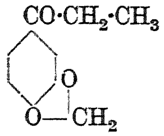
Anilide: cryst. M.p. 160°. Insol. H_2O.

Anhydro-oxime: $C_{10}H_9O_2N$. MW, 175. M.p. 116–17°.

Auwers, Heinze, *Ber.*, 1919, **52**, 590.
De Benneville, *J. Org. Chem.*, 1941, **6**, 462.

Propiophenone-β-carboxylic Acid.
See 1-Benzoylpropionic Acid.

Propiopiperone (3 : 4-*Methylenedioxypropiophenone*)



$C_{10}H_{10}O_3$ MW, 178

Needles. M.p. 39°. B.p. 153–4°/13 mm. D^{20} 1·210.

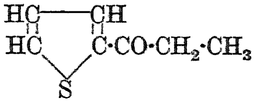
Oxime: prisms. M.p. 104°.

Semicarbazone: m.p. 187–8°.

Phenylhydrazone: yellow needles. M.p. 97°.

Foulds, Robinson, *J. Chem. Soc.*, 1914, **105**, 1972.
Schimmel, *Chem. Zentr.*, 1905, I, 1470.
Wallach, Pond, *Ber.*, 1895, **28**, 2719.

Propiothienone (2-*Propionylthiophene, ethyl 1-thienyl ketone*)



C_7H_8OS MW, 140

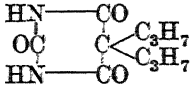
Pale yellow oil. B.p. 228°, 100–1°/11 mm. n_D^{20} 1·5539. Ox. ⟶ thiophene-2-carboxylic acid.

Oxime: plates. M.p. 55–6°.

Semicarbazone: plates from C_6H_6. M.p. 167°. Sol. hot H_2O, Me_2CO, C_6H_6. Spar. sol. Et_2O. Insol. pet. ether.

Steinkopf, Schubart, *Ann.*, 1921, **424**, 8.
Thomas, Couderc, *Bull. soc. chim.*, 1918, **23**, 288.
Hartough, Kosak, *J. Am. Chem. Soc.*, 1947, **69**, 3095.

Proponal (5 : 5-*Dipropylbarbituric acid*)



$C_{10}H_{16}O_3N_2$ MW, 212

Colourless plates from H_2O. M.p. 146° (166°). Aq. sol. has bitter taste. Sol. 1640 parts H_2O at 20°, 70 parts at 100°. Very sol. EtOH, Et_2O, AcOEt, $CHCl_3$, C_6H_6, alkalis. Forms mono-Na salt. Hypnotic.

Quinine comp.: needles. M.p. 127–8°. Very

sol. EtOH, Me_2CO. Sol. Et_2O, $CHCl_3$, hot H_2O, hot C_6H_6, hot ligroin.

Itallie, Steenhauer, *Pharm. Weekblad*, 1930, **67**, 977, (*Chem. Zentr.*, 1931, I, 823).
Merck, D.R.P., 249,908, (*Chem. Zentr.*, 1912, II, 777); D.R.P., 146,496, (*Chem. Zentr.*, 1903, II, 1483).
Conrad, *Ann.*, 1905, **340**, 321.
Fischer, Dilthey, *Ann.*, 1904, **335**, 344.

N-Propylacetamide.
See under Propylamine.

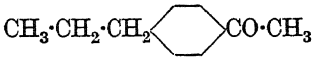
Propylacetanilide.
See under Aminopropylbenzene.

1-Propylacetoacetic Acid.
See 1-Aceto-*n*-valeric Acid.

Propylacetonylcarbinol.
See 4-Heptanolone-2.

Propyl acetonyl Ether.
See under Hydroxyacetone.

p-Propylacetophenone (p-*Acetopropylbenzene*)



$C_{11}H_{14}O$ MW, 162

Oil. B.p. 252°. D^{15} 0·9785. Ox. ⟶ *p*-propylbenzoic acid.
Oxime: plates from pet. ether. M.p. 43–4°.
Phenylhydrazone: yellowish-white hexagonal plates from pet. ether. M.p. 92°. Very sol. warm pet. ether. Very unstable.
Semicarbazone: m.p. 180–180·5°.

Widman, *Ber.*, 1888, **21**, 2224.

***n*-Propylacetylene.**
See 1-Pentyne.

2-Propylacrolein (2-*Hexenal*-1, 1-*hexylene aldehyde, hexenic aldehyde*)

$CH_3 \cdot CH_2 \cdot CH_2 \cdot CH{:}CH \cdot CHO$

$C_6H_{10}O$ MW, 98

Constituent of leaves of green plants. Colourless oil. B.p. 150°, 49·5°/18·5 mm. D_4^{13} 0·861. n_D^{13} 1·4470.
Semicarbazone: m.p. 175–6°.
p-*Nitrophenylhydrazone*: m.p. 139°.
2 : 4-*Dinitrophenylhydrazone*: m.p. 144°.

Delaby, Guillot-Allègre, *Bull. soc. chim.*, 1933, **53**, 301.
Curtius, Franzen, *Ann.*, 1914, **404**, 93; 1912, **390**, 89.

1-Propylacrylic Acid (*α-Amylene-β-carboxylic acid, 1-methylene-*n*-valeric acid, 1-pentene-2-carboxylic acid*)

$CH_3 \cdot CH_2 \cdot CH_2 \cdot C(=CH_2) \cdot COOH$

$C_6H_{10}O_2$ MW, 114

Prisms. M.p. 24·4°. B.p. 213°/750 mm, D_{25}^{25} 0·9812. Sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. H_2O. Heat of comb. C_v 795·7 Cal. $k = 0{\cdot}97 \times 10^{-5}$ at 25°. Gives spar. sol. Ca salt, cryst. Ag salt.
Et ester: $C_8H_{14}O_2$. MW, 142. B.p. 167–8°.

Lieben, Zeisel, *Monatsh.*, 1883, **4**, 46.

2-Propylacrylic Acid (1-*Hexenic acid*, 1 : 2-*hexenoic acid, α-amylene-α-carboxylic acid, 1-pentene-1-carboxylic acid, butylideneacetic acid, isohydrosorbic acid*)

$CH_3 \cdot CH_2 \cdot CH_2 \cdot CH{:}CH \cdot COOH$

$C_6H_{10}O_2$ MW, 114

Needles from H_2O or EtOH. M.p. 33° (36–7°). B.p. 216–17°, 118°/19 mm. $k = 1{\cdot}98 \times 10^{-5}$ at 25°. Br ⟶ 1 : 2-dibromocaproic acid. HBr ⟶ 2-bromocaproic acid. $AgClO_3$ ⟶ 1 : 2-dihydroxycaproic acid.
Me ester: $C_7H_{12}O_2$. MW, 128. B.p. 56–8°/13 mm. n_D^{19} 1·4601.
Et ester: b.p. 174–5°, 80°/22 mm. D_4^{20} 0·8986. n_D^{20} 1·4348.
p-*Bromophenacyl ester*: m.p. 62–62·5°.
Chloride: C_6H_9OCl. MW, 132·5. B.p. 70°/23 mm.
Anilide: needles. M.p. 109–10°.

Fittig, Baker, *Ann.*, 1894, **283**, 118.
Goldberg, Linstead, *J. Chem. Soc.*, 1928, 2351.
Boxer, Linstead, *J. Chem. Soc.*, 1931, 740.
Delaby, Guillot-Allègre, *Compt. rend.*, 1931, **192**, 1467.

1-Propyladipic Acid.
See Heptane-1 : 4-dicarboxylic Acid.

2-Propyladipic Acid

$CH_2 \cdot COOH$
$C_3H_7 \cdot CH$
CH_2
$CH_2 \cdot COOH$

$C_9H_{16}O_4$ MW, 188

Sinters at 45°. M.p. 49°. B.p. 184–6°/0·1 mm.
Dinitrile: $C_9H_{14}N_2$. MW, 150. B.p. 174–6°/12 mm.

v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 182.

Propyl Alcohol (*Propanol*-1)

$CH_3 \cdot CH_2 \cdot CH_2OH$

C_3H_8O MW, 60

B.p. 97·4°, 79–80°/374·6 mm., 65·94°/198·8 mm., 49·92°/90 mm., 30·35°/28·5 mm. D_4^{15} 0·80753, D_4^{20} 0·8035, D_4^{25} 0·7993. n_D^{20} 1·38499, n_D^{25} 1·3833. Misc. with H_2O, EtOH, Et_2O. Crit. temp. 263·7°. KOH at 240–50° ⟶ propionic acid + propylene. Br ⟶ 1 : 1-dibromopropionaldehyde + propyl bromide. I + KOH ⟶ iodoform.
Nitrite: *see* Propyl nitrite.
Nitrate: *see* Propyl nitrate.

Phosphate: *see under* Phosphoric Acid.
Acetyl: propyl acetate. B.p. 101·67°. D_4^{20} 0·88630, D_4^{40} 0·86390. n_D^{20} 1·38422.
Benzoyl: propyl benzoate. F.p. — 51·6°. B.p. 230·6–230·9°. D_4^{15} 1·0274. n_D^{15} 1·50139. Heat of comb. C_p 1255·010 Cal.
3 : 5-*Dinitrobenzoyl*: m.p. 73°.
Acid phthalate: m.p. 54–5°.
Phenylurethane: m.p. 58°.
p-*Nitrophenylurethane*: m.p. 115°.
1-*Naphthylurethane*: m.p. 105°.

Dreyfus, U.S.P., 1,996,101, (*Chem. Abstracts*, 1935, **29**, 3349).
du Pont, U.S.P., 2,014,740, (*Chem. Abstracts*, 1935, **29**, 7342).
Schüpphaus, *J. Am. Chem. Soc.*, 1892, **14**, 53.
Crismer, *Chem. Zentr.*, 1904, I, 1480.
Shriner, Cox, *J. Am. Chem. Soc.*, 1931, **53**, 1602.

Propylallene.
See 1 : 2-Hexadiene.

3-Propylallyl Alcohol.
See 2-Hexenol-1.

Propylallylamine

$CH_2{:}CH{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_6H_{13}N$ MW, 99

B.p. 110–14°. D^{18} 0·7708. Sol. 15–20 parts H_2O.

Liebermann, Paal, *Ber.*, 1883, **16**, (I), 526.

Propylallylcarbinol.
See 1-Heptenol-4.

Propyl allyl Ether

$CH_2{:}CH{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_6H_{12}O$ MW, 100

B.p. 90–2°. D^{20} 0·7764.

Deulofeu, *Chem. Zentr.*, 1928, II, 2547.
Lippert, *Ann.*, 1893, **276**, 192.

Propyl allyl Ketone.
See 1-Heptenone-4.

Propylamine (1-*Aminopropane*)

$CH_3{\cdot}CH_2{\cdot}CH_2NH_2$

C_3H_9N MW, 59

B.p. 49°. D_4^4 0·7330, D_4^{25} 0·714. $n_D^{16\cdot6}$ 1·39006, $n_D^{23\cdot5}$ 1·3873. Heat of comb. C_p (vapour) 575·74 Cal., C_p (liq.) 560·3 Cal., C_v (liq.) 559·4 Cal. $k = 4{\cdot}7 \times 10^{-4}$ at 25°.
B,HCl: m.p. 157–8°.
B,HAuCl₄: m.p. 169°.
B_2,H_2PtCl_6: m.p. 214°.
B_2,H_2PtBr_6: red leaflets. M.p. 257–8° decomp.
Picrate: m.p. 135°.
N-*Acetyl*: *N*-propylacetamide. $C_5H_{11}ON$. MW, 101. B.p. 222–5°. *B,HCl*: needles. M.p. 47°.
N-*Benzoyl*: *N*-propylbenzamide. Cryst. from EtOH or C_6H_6. M.p. 84·5°. B.p. 294–5°/750 mm. slight decomp.

Neogi, Chowdhuri, *J. Chem. Soc.*, 1917, **111**, 902.
Rakshit, *J. Am. Chem. Soc.*, 1913, **35**, 445.
Hofmann, *Ber.*, 1882, **15**, 769.
Linnemann, *Ann.*, 1872, **161**, 44.
Beregi, *Compt. rend.*, 1947, **224**, 1508.

2-Propylaminoethyl Alcohol (N-2-*Hydroxyethylpropylamine, propylethanolamine*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2OH$

$C_5H_{13}ON$ MW, 103

Oil. B.p. 182°/746 mm. D_4^{20} 0·9005. n_D^{20} 1·4428. Sol. H_2O, EtOH, Et_2O. Strongly basic.
B,HAuCl₄: plates from H_2O. M.p. 85°.
Picrate: yellowish-brown prisms from H_2O. M.p. 129°. Sol. H_2O, EtOH.
Picrolonate: brownish-red prisms from EtOH.Aq. M.p. 238° decomp.

Matthes, *Ann.*, 1901, **315**, 110.

γ-Propylaminopropylbenzene.
See under 3-Phenylpropylamine.

Propyl-*n*-amylcarbinol (*Nonanol*-4)

$CH_3{\cdot}[CH_2]_3{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_9H_{20}O$ MW, 144

B.p. 192–3°. D^{20} 0·8282. n_D 1·41971. Insol. H_2O.
Acetyl: b.p. 199–200°. D^{20} 0·8282.

Pexters, *Chem. Zentr.*, 1907, I, 1398.

***n*-Propyl-*tert*.-amylcarbinol.**
See 3 : 3-Dimethylheptanol-4.

Propyl *n*-amyl Ketone (*Nonanone*-4)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_9H_{18}O$ MW, 142

B.p. 187–8°, 75–6°/20 mm., 70·5–71·5°/10 mm. D_4^0 0·837.
Semicarbazone: cryst. from EtOH. M.p. 73–4° (rapid heat.) (67°, 145°).
p-*Nitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 84–5°.

Karrer, Wettstein, Fröwis, Morf, *Helv. Chim. Acta*, 1932, **15**, 231.
Bryant, Clemo, *J. Chem. Soc.*, 1931, 2080.

***N*-Propylaniline**

$C_6H_5{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_9H_{13}N$ MW, 135

Pale yellow oil. B.p. 222°. D^{18} 0·949.
Hydrochloride: needles. M.p. 150°.
Oxalate: plates from H_2O. M.p. 152°.

Voss, Blanke, *Ann.*, 1931, **485**, 280.
Hickinbottom, *J. Chem. Soc.*, 1930, 992.

Propylaniline.
See Aminopropylbenzene.

Propylanisole.
See under Propylphenol.

***N*-Propylbenzamide.**
See under Propylamine.

Propylbenzene (1-*Phenylpropane*)

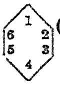
$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

C_9H_{12} MW, 120

M.p. −99·2°. B.p. 159·45°. D_0^0 0·87864, D_4^{20} 0·8617. n_D^{20} 1·4925. Heat of comb. C_v 1244·6 Cal. $K_2Cr_2O_7 + H_2SO_4 \longrightarrow$ benzoic acid.
Picrate: yellow cryst. M.p. 103·5°.

Gilman, Catlin, *Organic Syntheses*, Collective Vol. I, 458.
Auwers, *Ann.*, 1919, **419**, 92.
Baril, Hauber, *J. Am. Chem. Soc.*, 1931, **53**, 1087.

***o*-Propylbenzoic Acid**

COOH

$CH_2{\cdot}CH_2{\cdot}CH_3$

$C_{10}H_{12}O_2$ MW, 164

Leaflets from EtOH.Aq. M.p. 58°. B.p. 272°/739 mm.
Et ester: $C_{12}H_{16}O_2$. MW, 192. B.p. 244–7°/785 mm. D_{15}^{15} 1·003.
Chloride: $C_{10}H_{11}OCl$. MW, 182·5. Yellow liq. B.p. 236°.
Amide: $C_{10}H_{13}ON$. MW, 163. Needles from H_2O. M.p. 127–8°. Sol. EtOH, Et_2O, AcOH, C_6H_6.
Nitrile: $C_{10}H_{11}N$. MW, 145. B.p. 227–9°/758 mm.

Gabriel, Michael, *Ber.*, 1878, **11**, 1014.
Gottlieb, *Ber.*, 1899, **32**, 961.

***p*-Propylbenzoic Acid.**
Prisms or leaflets from H_2O. M.p. 141°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. boiling H_2O. Volatile in steam. $KMnO_4 \longrightarrow$ terephthalic acid.
Nitrile: b.p. 227°.

Widman, *Ber.*, 1889, **22**, 2278.
Francksen, *Ber.*, 1884, **17**, 1229.

α-Propylbenzyl Alcohol.
See Propylphenylcarbinol.

Propylbenzylcarbinol (1-*Phenylpentanol*-2, 1-*phenyl*-sec.-n-*amyl alcohol*, β-*hydroxy*-n-*amylbenzene*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}C_6H_5$

$C_{11}H_{16}O$ MW, 164

B.p. 127°/15 mm. D_4^{25} 0·9579. n_{5875}^{25} 1·51017.
Phenylurethane: needles from pet. ether. M.p. 80°.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

Propylbenzylethylene.
See 1-Phenyl-2-hexene.

Propyl benzyl Ketone (ω-*Butyryltoluene*, β-*ketoamylbenzene*, 1-*phenylpentanone*-2)

$C_6H_5{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_{11}H_{14}O$ MW, 162

B.p. 244°. D_4^0 0·984.
Semicarbazone: m.p. 84°.

Senderens, *Ann. chim.*, 1913, **28**, 321.
Ludlam, *J. Chem. Soc.*, 1902, **81**, 1189.

Propyl bromide (1-*Bromopropane*)

$CH_3{\cdot}CH_2{\cdot}CH_2Br$

C_3H_7Br MW, 123

B.p. 71°. Spar. sol. H_2O. D_4^{20} 1·3529. n_D^{20} 1·43414. Heat of comb. C_p 499·3 Cal. Heat with $AlBr_3 \longrightarrow$ isopropyl bromide.

Karvonen, *Chem. Abstracts*, 1920, **14**, 2176.
Tseng, Hou, *J. Chinese Chem. Soc.*, 1934, **2**, 57.
Bodroux, *Compt. rend.*, 1915, **160**, 205.
Norris, *Am. Chem. J.*, 1907, **38**, 640.
Goshorn, Boyd, Degering, *Organic Syntheses*, Collective Vol. I, (2*nd Ed.*), 36.

3-Propylbutanone.
See 3-Methylhexanone-2.

Propyl-*sec*.-*n*-butylacetic Acid.
See 2-Methyl-1-propyl-*n*-valeric Acid.

3-Propyl-*sec*.-*n*-butyl Alcohol.
See 3-Methylhexanol-2.

Propyl-*n*-butylcarbinol (*Octanol*-4, 4-*hydroxyoctane*)

$CH_3{\cdot}[CH_2]_3{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_8H_{18}O$ MW, 130

d-.
B.p. 79°/16 mm. D_4^{22} 0·818. n_D^{25} 1·4275. $[\alpha]_D^{22}$ + 0·74°.

dl-.
B.p. 71°/10 mm. D_4^0 0·838. n_D^{17} 1·4257.
α-*Naphthylurethane*: fluffy needles from pet. ether. M.p. 61–2°.

Levene, Marker, *J. Biol. Chem.*, 1931, **91**, 405.
Bouveault, Locquin, *Bull. soc. chim.*, 1906, **35**, 644, 646.
Levene, Kuna, *J. Biol. Chem.*, 1941, **140**, 264.

Propyl-*sec*.-*n*-butylcarbinol (3-*Methylheptanol*-4)

$$\begin{array}{c}\quad CH_3 \\ CH_3{\cdot}CH_2{\cdot}\overset{|}{C}H{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CH_3\end{array}$$

$C_8H_{18}O$ MW, 130

B.p. 164·7°/760 mm., 67·3°/20 mm. D_4^{25} 0·8335. n_D^{25} 1·4211. Solubility in H_2O, 1·880 gm. per litre.

Bridgman, *Chem. Zentr.*, 1933, II, 348.

Propyl-*tert*.-butylcarbinol.
See 2 : 2-Dimethylhexanol-3.

Propylbutylene.
See 3-Methyl-1-hexene *and* 3-Methyl-2-hexene.

Propyl *n*-butyl Ether

$CH_3{\cdot}[CH_2]_2{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_7H_{16}O$ MW, 116

B.p. 117·1°. D^0 0·7773.

Dobriner, *Ann.*, 1888, **243**, 7.

Propyl *tert.*-butyl Ether

$(CH_3)_3C{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_7H_{16}O$ MW, 116

B.p. 97·4°. D_4^{25} 0·7472. n_D^{25} 1·3830.

Norris, Rigby, *J. Am. Chem. Soc.*, 1932, **54**, 2088.

1-Propyl-2-butylethylene.
See 4-Nonene.

Propyl *n*-butyl Ketone (4-*Keto-octane, octanone*-4)

$CH_3{\cdot}[CH_2]_3{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_8H_{16}O$ MW, 128

B.p. 165–8°, 70°/26 mm. n_D^{14} 1·4173.
Oxime: b.p. 116–17°/20 mm.
2 : 4-*Dinitrophenylhydrazone*: orange needles from pet. ether. M.p. 41°.

Bouveault, Locquin, *Bull. soc. chim.*, 1906, **35**, 648.

Propyl *sec.*-*n*-butyl Ketone.
See 3-Methylheptanone-4.

2-Propylbutyraldehyde.
See 2-Methylcaproic Aldehyde.

2-Propylbutyric Acid.
See 2-Methylcaproic Acid.

Propylcarbamic Acid.
Et ester, *see* Propylurethane: Amide, *see* Propylurea.

Propylcarbylamine.
See Propyl isocyanide.

Propylcatechol.
See 2 : 3-Dihydroxy-1-propylbenzene *and* 3 : 4-Dihydroxy-1-propylbenzene.

Propyl chloride (1-*Chloropropane*)

$CH_3{\cdot}CH_2{\cdot}CH_2Cl$

C_3H_7Cl MW, 78·5

M.p. − 122·8°. B.p. 46·60°. D_4^{15} 0·89694, D_4^{20} 0·8910. n_D^{20} 1·38838. Heat of comb. C_p (vapour) 492·38 Cal.

Norris, Taylor, *J. Am. Chem. Soc.*, 1924, **46**, 756.
Dehn, Davis, *J. Am. Chem. Soc.*, 1907, **29**, 1329.
Norris, *Organic Syntheses*, Collective Vol. I, 138.

3-Propylcrotonaldehyde.
See 1-Heptenal.

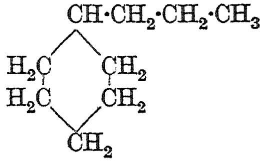
2-Propylcrotonic Acid.
See 2-Methyl-1-hexenic Acid.

Propyl cyanide.
See under *n*-Butyric Acid.

Propylcyanoacetic Acid.
See under Propylmalonic Acid.

1-Propyl-1-cyanoisovaleric Acid.
See under Propylisopropylmalonic Acid.

Propylcyclohexane (*Hexahydropropylbenzene*)

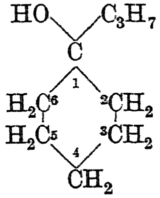


C_9H_{18} MW, 126

Liq. with odour resembling petrol. F.p. − 95·0°. B.p. 154·5–155·5°/756 mm. $D_4^{20\,vac.}$ 0·7898. n_D^{21} 1·437.

Eisenlohr, *Chem. Abstracts*, 1926, **20**, 171.
Bourguel, *Bull. soc. chim.*, 1927, **41**, 1475.

1-Propylcyclohexanol (1-*Propylhexahydrophenol*)



$C_9H_{18}O$ MW, 142

Liq. with odour resembling camphor. B.p. 180° decomp., 85°/20 mm. D_0^0 0·945, D_4^{12} 0·934. n_D^{12} 1·468. Insol. H_2O.
p-*Nitrobenzoyl*: obtained in very poor yield after a reaction period of two months. Cryst. from pet. ether. M.p. 46–8°.

Sabatier, Mailhe, *Bull. soc. chim.*, 1905, **33**, 75.
Hückel, Nerdel, Reimer, *J. prakt. Chem.*, 1937, **149**, 315.

2-Propylcyclohexanol (2-*Propylhexahydrophenol*).

Cis:
B.p. 84°/10 mm. D_4^{11} 0·9247. n_D^{11} 1·4688.
Isovaleryl: b.p. 138–9°/18 mm. D^{11} 0·914. n_D^{11} 1·450.
Acid succinate: cryst. from AcOH.Aq. M.p. 31–2°.
Benzoyl: b.p. 177–8°/14 mm. D_4^{16} 1·0262. n_D^{16} 1·5150.
3 : 5-*Dinitrobenzoyl*: needles from pet. ether. M.p. 75°.
Acid phthalate: cryst. from EtOH.Aq. M.p. 107–8°.

Phenylurethane: cryst. from pet. ether. M.p. 97–97·5°.
α-Naphthylurethane: m.p. 103–4°.

Trans:
B.p. 90°/14 mm. D_4^{11} 0·9160. n_D^{11} 1·4668.
Isovaleryl: b.p. 129–30°/13 mm. D_4^{11} 0·9131. n_D^{11} 1·4490.
Acid succinate: m.p. 48–9°.
Benzoyl: b.p. 179–80°/15 mm. D_4^{16} 1·0154. n_D^{16} 1·513.
Acid phthalate: plates from EtOH.Aq. M.p. 120–1°.
Phenylurethane: m.p. 69–70°.

Vavon, Anziani, *Bull. soc. chim.*, 1927, **41**, 1638.

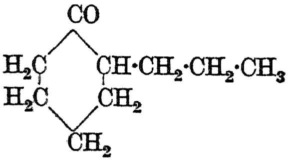
4-Propylcyclohexanol (*4-Propylhexahydrophenol*).

Cis:
B.p. 99°/17 mm. D_4^{16} 0·9102. $n_D^{16·5}$ 1·4652. More viscous than the trans isomer.
p-*Nitrobenzoyl*: needles or plates from EtOH. Aq. M.p. 89–89·5°.
Phenylurethane: cryst. from EtOH.Aq. M.p. 78–9°.
α-Naphthylurethane: cryst. from pet. ether. M.p. 92–92·8°.

Trans:
B.p. 208°/762 mm., 103°/14 mm. D_4^{17} 0·9072. $n_D^{17·5}$ 1·4613.
Acid phthalate: cryst. from pet. ether. M.p. 115–16°.
p-*Nitrobenzoyl*: cryst. from EtOH.Aq. M.p. 80–80·5°.
3 : 5-*Dinitrobenzoyl*: m.p. 124–5°.
Phenylurethane: cryst. from EtOH. M.p. 130–130·5°.
α-Naphthylurethane: fine needles from pet. ether. M.p. 134–5°.

Harris, D'Ianni, Adkins, *J. Am. Chem. Soc.*, 1938, **60**, 1469.
Ungnade, Ludutsky, *J. Org. Chem.*, 1945, **10**, 520.

2-Propylcyclohexanone



$C_9H_{16}O$ MW, 140

B.p. 198–9°/748 mm., 83–4°/13 mm. D_4^{13} 0·9145. n_D^{13} 1·4558.
Oxime: cryst. from EtOH.Aq. M.p. 67–8°.
Semicarbazone: cryst. from EtOH.Aq. M.p. 133·5–134° decomp.

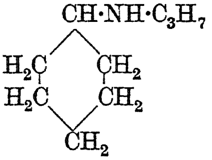
Vavon, Anziani, *Bull. soc. chim.*, 1927, **41**, 1638.

4-Propylcyclohexanone.

Liq. with disagreeable penetrating odour. B.p. 212°/740 mm., 97·5°/15 mm. D_4^{19} 0·9072. n_D^{19} 1·4530.
Oxime: cryst. from EtOH.Aq. M.p. 38–9°. B.p. 135–6°/15 mm. n_D^{14} 1·4902.
2 : 4-*Dinitrophenylhydrazone*: cryst. from EtOH. M.p. 132·5–133°.
Semicarbazone: cryst. from EtOH.Aq. M.p. 188·5° (179·5–180·5°).

Gauthier, *Compt. rend.*, 1944, **218**, 651.

N-Propylcyclohexylamine (*Hexahydro-N-propylaniline*)

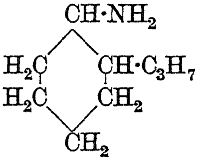


$C_9H_{19}N$ MW, 141

B.p. 185°.
Hydrochloride: m.p. 248–50°.
Phenylurea deriv.: needles from EtOH. M.p. 122–3°.

Skita, Keil, *Monatsh.*, 1929, **53** and **54**, 759.

2-Propylcyclohexylamine



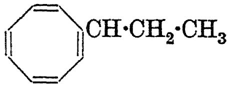
$C_9H_{19}N$ MW, 141

B.p. 60°/14 mm. D_4^{19} 0·8752.
N-*Benzenesulphonyl*: m.p. 131–2°.
Methiodide: m.p. 208–10°.

v. Braun, Bayer, *Ber.*, 1925, **58**, 390.

Propyl cyclohexyl Ketone.
See Hexahydrobutyrophenone.

Propylcyclo-octatetraene

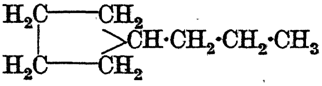


$C_{11}H_{12}$ MW, 144

Oil. B.p. 86–90°/18–20 mm., 73°/9 mm. D_4^{25} 0·8870. n_D^{25} 1·5131.
$AgNO_3$ add. comp.: pale yellow cryst. from EtOH. M.p. 141° decomp.

Cope, Van Orden, *J. Am. Chem. Soc.*, 1952, **74**, 175.
Cope, Campbell, *ibid.*, 179.

Propylcyclopentane

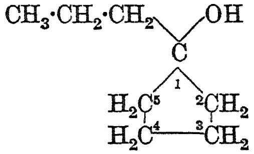


C_8H_{16} MW, 112

F.p. − 118·7°. B.p. 130·8°. D_4^{20} 0·7761. n_D^{20} 1·4266.

Chavanne, Becker, *Bull. soc. chim. Belg.*, 1927, **36**, 591.
Zelinskii, Kazanskii, *Chem. Abstracts*, 1935, **29**, 153.
Crane, Boord, Henne, *J. Am. Chem. Soc.*, 1945, **67**, 1237.

1-Propylcyclopentanol



$C_8H_{16}O$ MW, 128

M.p. − 37·5°. B.p. 173·5°/760 mm., 83°/20 mm. D_4^{25} 0·9040. n_D^{25} 1·4502.
p-*Nitrobenzoyl*: cryst. from pet. ether. M.p. 59·5°.
3 : 5-*Dinitrobenzoyl*: cryst. from pet. ether. M.p. 82°.
Allophanate: m.p. 178°.

Chavanne, Becker, *Bull. soc. chim. Belg.*, 1927, **36**, 591.
McLellan, Edwards Jr., *J. Am. Chem. Soc.*, 1944, **66**, 409.

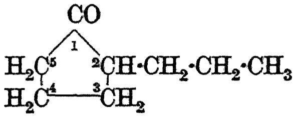
2-Propylcyclopentanol.

Cis:
B.p. 79–80°/12 mm. D_9^9 0·9165. n_D^9 1·4600.
Acid succinate: m.p. 27–8°.
3 : 5-*Dinitrobenzoyl*: cryst. from EtOH. M.p. 70–1°.
Acid phthalate: needles. M.p. 95–6°.
Phenylurethane: m.p. 83–4°.

Trans:
B.p. 78–9°/10 mm. D_9^9 0·9018. n_D^9 1·4565.
Acid succinate: viscous oil. D_{16}^{16} 1·0686. n_D^{16} 1·4605.
3 : 5-*Dinitrobenzoyl*: plates from EtOH. M.p. 30–1°.
Acid phthalate: cryst. from EtOH.Aq. M.p. 68°.
Phenylurethane: cryst. from EtOH.Aq. M.p. 61–2°.

Vavon, Flurer, *Bull. soc. chim.*, 1929, **45**, 756.

2-Propylcyclopentanone



$C_8H_{14}O$ MW, 126

B.p. 183·1–183·2°, 70·2°/15 mm., 59·4°/8 mm. D_4^{20} 0·9017. n_D^{20} 1·4429.
Oxime: b.p. 109–11°/9 mm.
Semicarbazone: cryst. from EtOH. M.p. 214° decomp.

Chiurdoglu, *Bull. soc. chim. Belg.*, 1934, **43**, 35.
Vavon, Flurer, *Bull. soc. chim.*, 1929, **45**, 754.

3-Propylcyclopentanone.
B.p. 190–1°. D_4^{20} 0·9041. n_D^{12} 1·4456.
Oxime: b.p. 121–2°/12 mm.
Semicarbazone: cryst. from MeOH. M.p. 178–9°.

v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 181.

***S*-Propylcysteine *S*-oxide.**
See Dihydroalliin.
Propyldichloroamine.
See *N*-Dichloropropylamine.
α-Propyldiphenylmethane.
See 1 : 1-Diphenyl-*n*-butane.
Propylene (*Propene*)

$CH_3{\cdot}CH{:}CH_2$

C_3H_6 MW, 42

Colourless gas. M.p. − 185·2°. B.p. − 47·8°/750 mm. n_{5876} 1·001. Crit. temp. 92·1°. Liquefies under 7–8 atms. press. Heat of comb. C_p 499·3 Cal. Abs. EtOH absorbs 12–13 vols. Absorbed quantitatively by solutions of mercuric nitrate and sulphate.
Nitrosite: prisms. M.p. 119–20°. Sn + HCl ⟶ propylenediamine.

Ipat'ev, *Ber.*, 1934, **67**, 1061.
Goudet, Schenker, *Helv. Chim. Acta*, 1927, **10**, 135.
Rosenthal, U.S.P., 1,939,084, (*Chem. Abstracts*, 1934, **28**, 1364).
Maximoff, U.S.P., 1,870,859, (*Chem. Zentr.*, 1932, II, 3013).
Senderens, Aboulenc, *Chem. Zentr.*, 1936, I, 3818.

Propylene bromide.
See 1 : 2-Dibromopropane.
Propylene bromohydrin.
See 1-Bromoisopropyl Alcohol.
Propylene-carboxylic Acid.
See Crotonic Acid, Isocrotonic Acid, 1-Methylacrylic Acid *and* Vinylacetic Acid.
Propylene chloride.
See 1 : 2-Dichloropropane.
Propylene chlorohydrin.
See 2-Chloropropyl Alcohol *and* 1-Chloroisopropyl Alcohol.
Propylene chloroiodide.
See 2-Chloro-1-iodopropane.
Propylene cyanide.
See under Methylsuccinic Acid.
Propylene-1 : 3-dialdehyde.
See Glutacondialdehyde.

Propylenediamine (1 : 2-*Diaminopropane*)

$CH_3 \cdot CH(NH_2) \cdot CH_2NH_2$

$C_3H_{10}N_2$ MW, 74

d-.

B.p. 120·5°. D_4^{25} 0·8584. $[\alpha]_D^{25}$ + 29·78°.

Di-l-*tartrate*: m.p. 143°.

l-.

B.p. 120·5°. D_4^{25} 0·8588. Sol. H_2O, $CHCl_3$. Insol. Et_2O. $[\alpha]_D^{25}$ − 29·70°. ($[\alpha]_D^{24\cdot3}$ − 20·957°).

B,2HCl: m.p. 240°. $[\alpha]_D^{25}$ − 4·04° in H_2O.

Di-d-*tartrate*: m.p. 143°.

Picrate: yellow cryst. M.p. 237°.

dl-.

B.p. 119–20°. D^{15} 0·878. Forms hydrate with $\frac{1}{2}H_2O$.

B,2HCl: m.p. 220°.

1-N-*Di-Et*: $C_7H_{18}N_2$. MW, 130. B.p. 152°.

1 : 2-N-*Diacetyl*: needles from C_6H_6. M.p. 138–9°. B.p. 190°/18 mm. Very sol. H_2O, EtOH, $CHCl_3$. Spar. sol. C_6H_6. Insol. Et_2O, ligroin.

1 : 2-N-*Dipropionyl*: needles. M.p. 165°. B.p. 190°/56 mm.

1 : 2-N-*Dibenzoyl*: needles from C_6H_6. M.p. 192–3°.

Dipicrate: yellow needles from H_2O. M.p. 137°.

Windaus, Dorries, Jensen, *Ber.*, 1921, **54**, 2750.
I.G., D.R.P., 551,436, (*Chem. Zentr.*, 1932, II, 740).
Hofmann, *Ber.*, 1873, **6**, 308.
Tsugajew, Sokolow, *Ber.*, 1909, **42**, 56.
Darzens, *Compt. rend.*, 1939, **208**, 1503.

Propylene dibromide.

See 1 : 2-Dibromopropane.

Propylene-dicarboxylic Acid.

See Glutaconic Acid *and* Itaconic Acid.

Propylene dichloride.

See 1 : 2-Dichloropropane.

Propylene Dithioglycol (1 : 2-*Dimercaptopropane, dithiopropylene glycol, propane*-1 : 2-*dithiol*)

$CH_3 \cdot CH(SH) \cdot CH_2SH$

$C_3H_8S_2$ MW, 108

Oil. B.p. 152°. Anti-arsenical.

Peters *et al.*, B.P. 579,971, (*Chem. Abstracts*, 1947, **41**, 2077).

Propylene Glycol (1 : 2-*Dihydroxypropane*, *α-propylene glycol*)

$CH_3 \cdot CH(OH) \cdot CH_2OH$

$C_3H_8O_2$ MW, 76

d-.

B.p. 95°/15 mm. $[\alpha]_D^{18}$ + 13·71° in H_2O. Readily racemises in H_2O.

Dibutyryl: b.p. 95–105°/15 mm. $[\alpha]_D^{18}$ + 2·05°.

l-.

B.p. 85–91°/12 mm. $[\alpha]_D^{20}$ − 9·8° in H_2O.

Di-phenylurethane: m.p. 146–7°. $[\alpha]_D^{20}$ + 13·3° in EtOH.

dl-.

Viscous oil with sweet taste. B.p. 188–9°, 96–8°/21 mm. $D^{19\cdot4}$ 1·0403. Misc. with H_2O and EtOH in all proportions. Conc. HI ⟶ isopropyl iodide. $ZnCl_2$ or 50% H_2SO_4 ⟶ propionaldehyde. H_2O_2 + $FeSO_4$ ⟶ acetone + propionaldehyde.

Diacetyl: b.p. 190–1°/762 mm. D_4^{20} 1·059. n_D^{20} 1·4173.

Di-p-*nitrobenzoyl*: cryst. from EtOH. M.p. 126–7°.

Di-phenylurethane: m.p. 152·5–153·5°.

Dinitrite: b.p. 108–10°. Insol. H_2O.

Monopalmitate: plates from EtOH. M.p. 54·3°.

Dipalmitate: plates from EtOH. M.p. 68·8°.

Monostearate: plates from EtOH. M.p. 59·5°.

Distearate: plates from EtOH. M.p. 72·3°.

1-*Me ether*: $C_4H_{10}O_2$. MW, 90. B.p. 126–7°. D_4^{20} 0·9260. n_D^{20} 1·4070. *Acetyl*: b.p. 147°/762 mm. D_4^{20} 0·9709. n_D^{20} 1·4045.

1-*Et ether*: $C_5H_{12}O_2$. MW, 104. B.p. 136°. D_4^{20} 0·9028. n_D^{20} 1·4. *Acetyl*: b.p. 158–60°. D_4^{20} 0·9461. n_D^{20} 1·4097.

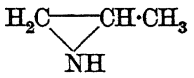
2-*Et ether*: b.p. 140–1°. D_4^{20} 0·9044. n_D^{20} 1·4122. Sol. H_2O.

Morley, Green, *J. Chem. Soc.*, 1885, **47**, 132.
Nef, *Ann.*, 1904, **335**, 291.
Ochiai, Miyaki, *Biochem. Z.*, 1935, **282**, 293.
Klebanskiĭ, Dolgopol'skiĭ, *Chem. Abstracts*, 1935, **29**, 5814.
du Pont, U.S.P., 1,963,997, (*Chem. Zentr.*, 1934, II, 3314).
Matignon, Moureu, Dodé, *Chem. Zentr.*, 1933, II, 646.
Levene, Walti, *J. Biol. Chem.*, 1926, **68**, 415.
Dewael, *Bull. soc. chim. Belg.*, 1930, **39**, 395.
Abderhalden, Eichwald, *Ber.*, 1918, **51**, 1319.
Howe, *Chem. Abstracts*, 1919, **13**, 1843.

β-Propylene Glycol.

See Trimethylene Glycol.

Propyleneimine



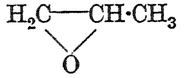
C_3H_7N MW, 57

Oil. B.p. 66–7°/751 mm. D^{16} 0·812. Fumes in air. Decomp. in aq. or HCl sol. Evaporate with HCl ⟶ 2-chloroisopropylamine.

Gabriel, v. Hirsch, *Ber.*, 1896, **29**, 2747.
Gabriel, Ohle, *Ber.*, 1917, **50**, 815.

Propylene iodohydrin.
See 1-Iodoisopropyl Alcohol.

Propylene oxide



C_3H_6O MW, 58

dl-.

Liq. with odour resembling ether. B.p. 35°. D^0 0·859. Misc. with H_2O, EtOH, Et_2O. Al_2O_3 at 250–60° ⟶ propionaldehyde + a little acetone. Ag_2O ⟶ acetic acid. NaHg + H_2O ⟶ isopropyl alcohol. Hot H_2O ⟶ propylene glycol. C_2H_5MgI in Et_2O ⟶ methylpropylcarbinol.

d-.

B.p. 36·5–38°. $[\alpha]_D^{18}$ + 12·72°. Part. racemises with hot H_2O.

l-.

$[\alpha]_D^{18}$ − 8·26°. Part. racemises with hot H_2O.

I.G., B.P. 292,066, (*Chem. Abstracts*, 1929, **23**, 1415).
Abderhalden, Eichwald, *Ber.*, 1918, **51**, 1318.
Krassuski, *Chem. Zentr.*, 1902, II, 19.
Henry, *Chem. Zentr.*, 1903, II, 486.

Propylene oxide carboxylic Acid.
See Methylglycidic Acid.

Propylene-tricarboxylic Acid.
See Aconitic Acid *and* Isaconitic Acid.

Propylethanolamine.
See Propylaminoethyl Alcohol.

Propyl *p*-ethoxyphenyl Ketone.
See under *p*-Hydroxybutyrophenone.

Propylethylene.
See 1-Pentene.

Propylethynylcarbinol.
See 1-Hexynol-3.

Propyl ethynyl Ketone.
See 1-Hexynone-3.

Propyl fluoride (1-*Fluoropropane*)

$CH_3·CH_2·CH_2F$

C_3H_7F MW, 62

Gas. M.p. − 159°. B.p. 3·2°/760 mm. n_D^{-20} 1·3326. Burns with bright flame.

Meslans, *Ann. chim. phys.*, 1894, **1**, 363.
du Pont, B.P. 406,284, (*Chem. Zentr.*, 1934, II, 132).
Grosse, Linn, *J. Org. Chem.*, 1938, **3**, 26.

Propylfumaric Acid (1-*Pentene*-1 : 2-*dicarboxylic acid*, *α-amylene-αβ-dicarboxylic acid*, *ethylmesaconic acid*)

$CH_3·CH_2·CH_2·C·COOH$
$HOOC·CH$ (C=CH double bond)

$C_7H_{10}O_4$ MW, 158

Leaflets. M.p. 174–5° (172·5–173°). Very sol. EtOH, Et_2O. Spar. sol. $CHCl_3$. Very spar. sol. C_6H_6, CS_2. Sol. 90 parts H_2O at ord. temp. $k = 9·3 \times 10^{-4}$ at 25°. CH_3COCl at 105–10° ⟶ ethylcitraconic anhydride.

Et ester-amide: $C_9H_{15}O_3N$. MW, 185. Needles. M.p. 78–9°.

Diamide: $C_7H_{12}O_2N_2$. MW, 156. M.p. 214–15° decomp.

Demarçay, *Ann. chim. phys.*, 1880, **20**, 489.
Walden, *Ber*, 1891, **24**, 2035.
Ssemenow, *Chem. Zentr.*, 1899, I, 783.

1-Propylglutaric Acid (*Hexane*-1 : 3-*dicarboxylic acid*)

$CH_3·CH_2·CH_2·CH·COOH$
CH_2
$CH_2·COOH$

$C_8H_{14}O_4$ MW, 174

Cryst. from H_2O. M.p. 66–8°. $k = 5·86 \times 10^{-5}$ at 24·4°.

Mellor, *J. Chem. Soc.*, 1901, **79**, 129.

2-Propylglutaric Acid

$CH_2·COOH$
$CH_3·CH_2·CH_2·CH$
$CH_2·COOH$

$C_8H_{14}O_4$ MW, 174

Needles from HCl. M.p. 52°. Sol. H_2O and most org. solvents. k (first) = $0·487 \times 10^{-4}$ at 25°: (second) = $4·11 \times 10^{-6}$ at 25°.

Di-Et ester: $C_{12}H_{22}O_4$. MW, 230. B.p. 132°/10 mm.

Anhydride: $C_8H_{12}O_3$. MW, 156. Mobile oil. B.p. 180°/20 mm.

Diamide: m.p. 195°.

Monoanilide: plates from C_6H_6. M.p. 128°.

Dianilide: cryst. from EtOH. M.p. 219°.

Day, Thorpe, *J. Chem. Soc.*, 1920, **117**, 1471.
Jeffery, Vogel, *J. Chem. Soc.*, 1939, 446.

1-Propylglyceric Acid (1 : 2-*Dihydroxypentane-2-carboxylic acid*)

C_3H_7
$HO·CH_2·C(OH)·COOH$

$C_6H_{12}O_4$ MW, 148

M.p. 94–5°. Sol. Et_2O.

Ssemenow, *Chem. Zentr.*, 1899, I, 1071.

2-Propylglyceric Acid (1 : 2-*Dihydroxycaproic acid*, 1 : 2-*dihydroxypentane*-1-*carboxylic acid*)

$CH_3·CH_2·CH_2·CH(OH)·CH(OH)·COOH$

$C_6H_{12}O_4$ MW, 148

Exists in two forms:

(i) Cryst. from AcOEt. M.p. 108·5°. Sol. hot H_2O. Mod. sol. Et_2O. Insol. $CHCl_3$.

Phenylhydrazide: m.p. 141·5–142°.

(ii) Cryst. from AcOEt. M.p. 99·5°. Sol. EtOH, hot H_2O. Mod. sol. Et_2O. Insol. $CHCl_3$.

Phenylhydrazide: m.p. 120–1°.

Braun, *J. Am. Chem. Soc.*, 1930, **52**, 3190.

1-Propylglycerol.
See Hexantriol-1 : 2 : 3.

Propylglyoxal (*1-Ketovaleraldehyde, 1-pentanalone-2*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CHO$

$C_5H_8O_2$ MW, 100

Greenish liq. with strong odour. B.p. 36°/16 mm. Reduces $NH_3.AgNO_3$. Colours Schiff's reagent. Polymerises.

Disemicarbazone: plates + 1AcOH from AcOH.Aq. M.p. 250°.

Osazone: needles from AcOH. M.p. 105°.

Di-Me acetal: $C_7H_{14}O_3$. MW, 146. B.p. 65–6°/14 mm. Reduces $NH_3.AgNO_3$ but not Fehling's.

Blaise, *Compt. rend.*, 1922, **175**, 1216.

Propylguaiacol.
See Cœrulignol *and under* 2 : 3-Dihydroxy-1-propylbenzene.

1-Propylguanidine

$H_2N{\cdot}C({=}NH){\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_4H_{11}N_3$ MW, 101

B_2,H_2SO_4: m.p. 220° decomp.

B,HNO_3: white cryst. M.p. 93°. Very sol. H_2O, EtOH.

$B,HAuCl_4$: red needles from EtOH. M.p. 200° decomp.

B_2,H_2PtCl_6: yellow prisms. M.p. 195° decomp.

Picrate: yellow needles from EtOH. M.p. 177–8°.

Piovano, *Gazz. chim. ital.*, 1928, **58**, 245.

4-Propyl-1-heptenol-4.
See Dipropylallylcarbinol.

Propyl heptyl Ketone (*Undecanone-4*)

$CH_3{\cdot}[CH_2]_5{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_{11}H_{22}O$ MW, 170

Oil with characteristic fruity odour. M.p. 4–5°. B.p. 106–7°/13 mm. D^{25} 0·8274. n_D^{24} 1·4248.

Oxime: b.p. 141°/13 mm.

Semicarbazone: plates from EtOH. M.p. 54–6°.

v. Braun, Kröper, *Ber.*, 1929, **62**, 2882.

Propylhexahydrocresol.
See Methylpropylcyclohexanol.

Propylhexahydrophenol.
See Propylcyclohexanol.

Propylhexahydrotoluene.
See Methylpropylcyclohexane.

3-Propylhexanol-3.
See Ethyldipropylcarbinol.

Propyl hexyl Ketone (*Decanone-4*)

$CH_3{\cdot}[CH_2]_5{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_{10}H_{20}O$ MW, 156

M.p. − 9°. B.p. 206–7°, 87–9°/11 mm. $D_0^{20.5}$ 0·824. Does not form bisulphite comp.

Semicarbazone: m.p. 51–2°.

Phenylhydrazone: amber oil. B.p. 197°/12 mm. n_D^{22} 1·5328.

Karrer, Shibata, Wettstein, Jacubowicz, *Helv. Chim. Acta*, 1930, **13**, 1300.

Propylhydrazine

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}NH_2$

$C_3H_{10}N_2$ MW, 74

B.p. 119°. Reduces $NH_3.AgNO_3$ and Fehling's in the cold.

Stolle, Bernath, *J. prakt. Chem.*, 1904, **70**, 280.

Propylhydroquinone.
See 2 : 5-Dihydroxy-1-propylbenzene.

Propyl 2-hydroxyisopropyl Ketone.
See 2-Methyl-2-hexanolone-3.

Propylhydroxylamine (*1-Hydroxylaminopropane*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}OH$

C_3H_9ON MW, 75

Needles from Et_2O. M.p. 77° (about 46°). Very volatile. Spar. sol. ligroin. Reacts basic. Reduces Fehling's.

Pierron, *Bull. soc. chim.*, 1899, **21**, 784.
Kjellin, *Ber.*, 1897, **30**, 1892.

Propyl hydroxynaphthyl Ketone.
See Hydroxybutyronaphthone.

Propyl hydroxyphenyl Ketone.
See o-, m-, *and* p-Hydroxybutyrophenone.

Propyl *p*-hydroxyphenyl sulphide.
See under Thiohydroquinone.

Propyl 1-hydroxypropyl Ketone.
See 3-Heptanolone-4.

Propyl hydroxytolyl Ketone.
See Hydroxy-methylbutyrophenone.

Propylideneacetic Acid.
See 2-Ethylacrylic Acid.

Propylideneacetone.
See 3-Hexenone-2.

Propylideneaniline.
See under Propionaldehyde.

2-Propylidenebutane.
See 3-Methyl-3-hexene.

2-Propylidene-*n*-butyl Alcohol.
See 3-Methyl-3-hexenol-1.

Propylidenebutylene.
See 1 : 4-Heptadiene, 2 : 4-Heptadiene *and* 3-Methyl-1 : 3-pentadiene.

2-Propylidenebutyric Acid.
See 2-Methylhydrosorbic Acid.

3-Propylidenebutyric Acid.
See 3-Heptenic Acid.

Propylidene chloride.
See 1 : 1-Dichloropropane.

Propylidene chlorobromide.
See 1-Chloro-1-bromopropane.

α-Propylidenediphenylmethane.
See 1 : 1-Diphenyl-1-butylene.

2-Propylidene-ethyl Alcohol (2-*Pentenol*-1, 3-*ethylallyl alcohol*, *α-butenylcarbinol*)

$CH_3{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CH_2OH$

$C_5H_{10}O$ MW, 86

Liq. B.p. 141–2° (138–9°). D_4^0 0·8645, D_4^{20} 0·8468. n_D^{20} 1·4299.

Acetyl : b.p. 149–51°. D^{22} 0·9019. n_D^{22} 1·4219.

Bouis, *Ann. chim.*, 1928, **9**, 402.
Prévost, *Ann. chim.*, 1928, **10**, 113.
Delaby, *Ann. chim.*, 1923, **20**, 196.

Propylidene-ethylene.
See 1 : 2-Pentadiene.

3-Propylideneisobutane.
See 2-Methyl-3-hexene.

1-Propylideneisobutyric Acid.
See 1-Methylhydrosorbic Acid.

2-Propylidenepropane.
See 2-Methyl-2-pentene.

2-Propylidenepropionaldehyde.
See 1-Methyl-2-ethylacrolein.

1-Propylidenepropionic Acid.
See 1-Methyl-2-ethylacrylic Acid.

2-Propylidenepropionic Acid.
See Hydrosorbic Acid.

Propylidenepropyl Alcohol.
See 3-Hexenol-1 *and* 2-Methyl-2-pentenol-1.

3-Propylidene-propylene.
See 1 : 3-Hexadiene.

Propylidenesuccinic Acid (*Ethylitaconic acid*, 2-*pentene*-1 : 2-*dicarboxylic acid*)

$CH_3{\cdot}CH_2{\cdot}CH{:}C{\cdot}COOH$
$\qquad\qquad\qquad\quad |$
$\qquad\qquad\qquad CH_2{\cdot}COOH$

$C_7H_{10}O_4$ MW, 158

Prisms from H_2O. M.p. 164–5° (163–7° decomp.). Spar. sol. $CHCl_3$, cold H_2O. Very spar. sol. Et_2O, C_6H_6, ligroin. $k = 3{\cdot}56 \times 10^{-5}$ at 25°.

Fichter, Probst, *Ann.*, 1910, **372**, 76.

Propyl iodide (1-*Iodopropane*)

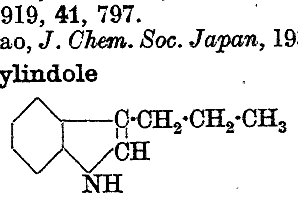
$CH_3{\cdot}CH_2{\cdot}CH_2I$

C_3H_7I MW, 170

B.p. 102·5°. D_4^{20} 1·7471. n_D^{20} 1·50546. Heat of comb. C_p 514·3 Cal., C_v 512·3 Cal.

Adams, Voorhees, *J. Am. Chem. Soc.*, 1919, **41**, 797.
Hirao, *J. Chem. Soc. Japan*, 1931, **52**, 269.

3-Propylindole



$C_{11}H_{13}N$ MW, 159

B.p. 162–4°/20 mm.

Picrate : red needles from pet. ether–C_6H_6. M.p. 113–14°.

Cornforth, Robinson, *J. Chem. Soc.*, 1942, 681.

1-Propylisoamyl Alcohol.
See Propylisobutylcarbinol.

Propylisoamylamine

$(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_8H_{19}N$ MW, 129

Liq. B.p. 148–9° (141°).

Sabatier, Mailhe, *Compt. rend.*, 1909, **148**, 900.
Freundler, Juillard, *ibid.*, 290.

Propylisoamylcarbinol (2-*Methyloctanol*-5, 1-*propylisohexyl alcohol*)

$(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_9H_{20}O$ MW, 144

B.p. 184–6°. D_4^0 0·8335, D_4^{15} 0·8199.

Douris, *Compt. rend.*, 1913, **157**, 57.

Propyl isoamyl Ketone (2-*Methyloctanone*-5)

$(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_9H_{18}O$ MW, 142

B.p. 177–9°. D_4^0 0·8362, D_4^{20} 0·8239. n_D^{20} 1·4210.

Semicarbazone : m.p. 144·5–145·5°.

Douris, *Compt. rend.*, 1913, **157**, 56.
Powell, Hagemann, *J. Am. Chem. Soc.*, 1944, **66**, 372.

Propylisobutylacetic Acid.
See 1-Propylisocaproic Acid.

1-Propylisobutyl Alcohol.
See Propylisopropylcarbinol.

Propylisobutylamine

$(CH_3)_2CH{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_7H_{17}N$ MW, 115

Liq. with odour resembling fusel-oil. B.p. 123–5°. Spar. sol. H_2O.

B,HCl : plates from EtOH–Et_2O. M.p. 135°.
Acid oxalate : needles. M.p. 224°.
B,HAuCl$_4$: m.p. 187–8°. Very sol. EtOH.
B$_2$,H$_2$PtCl$_6$: orange cryst. M.p. 187–8°. Sol. EtOH, hot H_2O.

Paal, Heupel, *Ber.*, 1891, **24**, 3048.
Pope, Read, *J. Chem. Soc.*, 1912, **101**, 522.

Propylisobutylcarbinol (2-*Methylheptanol*-4, 1-*propylisoamyl alcohol*)

$(CH_3)_2CH{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_8H_{18}O$ MW, 130

Somewhat viscous pleasant-smelling liq. B.p. 164° (160°). D^{20} 0·8207. n_D 1·42031. Sol. most org. solvents. Insol. H_2O.

Clarke, *J. Am. Chem. Soc.*, 1909, **31**, 114.
Muset, *Chem. Zentr.*, 1907, I, 1313.

Propyl isobutyl Ether

$$(CH_3)_2CH\cdot CH_2\cdot O\cdot CH_2\cdot CH_2\cdot CH_3$$

$C_7H_{16}O$ MW, 116

B.p. 106°/720 mm. D^{15} 0·7549. $n_{5461}^{24\cdot 9}$ 1·3852. Very spar. sol. H_2O.

Henstock, *J. Chem. Soc.*, 1931, 371.

Propyl isobutyl Ketone (*2-Methylheptanone-4*)

$$(CH_3)_2CH\cdot CH_2\cdot CO\cdot CH_2\cdot CH_2\cdot CH_3$$

$C_8H_{16}O$ MW, 128

B.p. 155°/750 mm. D_0^{22} 0·813. Does not form bisulphite comp.

Semicarbazone: m.p. 123–4°.

Phenylhydrazone: light yellow oil. B.p. 172°/16 mm. $n_D^{13\cdot 2}$ 1·5455.

Fournier, *Bull. soc. chim.*, 1910, **7**, 839.

Propylisobutylmalonic Acid (*2-Methylheptane-4 : 4-dicarboxylic acid*)

$$\begin{array}{r}CH_3\cdot CH_2\cdot CH_2\\(CH_3)_2CH\cdot CH_2\end{array}\!>C<\!\begin{array}{l}COOH\\COOH\end{array}$$

$C_{10}H_{18}O_4$ MW, 202

Prisms or plates from H_2O. M.p. 147–9° decomp. Sol. EtOH, Et_2O. Spar. sol. C_6H_6, cold H_2O.

Di-Et ester: $C_{14}H_{26}O_4$. MW, 258. B.p. 126°/9·5 mm.

Fischer, Holzapfel, v. Gwinner, *Ber.*, 1912, **45**, 252.

1-Propylisocaproic Acid (*2-Methylheptane-4-carboxylic acid, propylisobutylacetic acid*)

$$(CH_3)_2CH\cdot CH_2\cdot\overset{C_3H_7}{\overset{|}{C}}H\cdot COOH$$

$C_9H_{18}O_2$ MW, 158

d-.

Oil. B.p. about 100°/0·5 mm. D^{22} 0·8876. $[\alpha]_D^{22}+9\cdot 80°$.

dl-.

B.p. 125–7°/12 mm., 122°/8·5 mm. D^{20} 0·8928. Sol. EtOH, Et_2O. Very spar. sol. H_2O.

Fischer, Holzapfel, v. Gwinner, *Ber.*, 1912, **45**, 253.

Propyl isocyanide (*Propylcarbylamine*)

$$CH_3\cdot CH_2\cdot CH_2\cdot NC$$

C_4H_7N MW, 69

B.p. 99·5°. Heat of comb. C_p 638·9 Cal.

Guillemard, *Ann. chim. phys.*, 1908, **14**, 412.

1-Propylisohexyl Alcohol.

See Propylisoamylcarbinol.

Propylisopropylacetic Acid.

See 2-Methylhexane-3-carboxylic Acid.

Propylisopropylcarbinol (*2-Methylhexanol-3, 1-propylisobutyl alcohol*)

$$(CH_3)_2CH\cdot CH(OH)\cdot CH_2\cdot CH_2\cdot CH_3$$

$C_7H_{16}O$ MW, 116

d-.

B.p. 145–6°. $D_4^{16\cdot 7}$ 0·8266. $[\alpha]_D^{20}+21\cdot 25°$.

Acid phthalate: m.p. 79–80°. $[\alpha]_D + 8\cdot 36°$ in EtOH.

dl-.

Pleasant-smelling liq. with burning taste. B.p. 141–2°/765 mm. D^{17} 0·821. n_D 1·41493. Insol. H_2O.

Acid phthalate: m.p. 59–60°.

Muset, *Chem. Zentr.*, 1907, I, 1313.
Pickard, Kenyon, *J. Chem. Soc.*, 1912, **101**, 633.

Propyl isopropyl Ether

$$(CH_3)_2CH\cdot O\cdot CH_2\cdot CH_2\cdot CH_3$$

$C_6H_{14}O$ MW, 102

B.p. 83°. D_4^0 0·7597, $D_4^{12\cdot 5}$ 0·7474. n_D^{21} 1·376.

Bennett, Philip, *J. Chem. Soc.*, 1928, 1930.
Truchet, Graves, *Bull. soc. chim.*, 1932, **51**, 686.

Propyl isopropyl Ketone (*2-Methylhexanone-3*)

$$(CH_3)_2CH\cdot CO\cdot CH_2\cdot CH_2\cdot CH_3$$

$C_7H_{14}O$ MW, 114

Oil with peppermint odour. B.p. 135–6° (129–30°).

Semicarbazone: m.p. 119°.

Meerwein, *Ann.*, 1919, **419**, 138.

Propylisopropylmalonic Acid (*2-Methylhexane-3 : 3-dicarboxylic acid*)

$$\begin{array}{r}CH_3\cdot CH_2\cdot CH_2\\(CH_3)_2CH\end{array}\!>C<\!\begin{array}{l}COOH\\COOH\end{array}$$

$C_9H_{16}O_4$ MW, 188

d-.

Mononitrile: cryst. M.p. 94–5°. $[\alpha]_D^{20}+11\cdot 5°$ in toluene.

l-.

Mononitrile: cryst. from Et_2O. M.p. 90°. $[\alpha]_D^{20}-10\cdot 5°$ in toluene.

dl-.

Di-Et ester: $C_{13}H_{24}O_4$. MW, 244. B.p. 143°/42 mm. D_{25}^{25} 0·9803. n^{25} 1·4239.

Monoamide: $C_9H_{17}O_3N$. MW, 187. Prisms from H_2O. M.p. 137° decomp. Sol. EtOH, Me_2CO. Spar. sol. ligroin, cold H_2O.

Mononitrile: 1-isopropyl-1-cyanovaleric acid, 1-propyl-1-cyanoisovaleric acid. $C_9H_{15}O_2N$. MW, 169. M.p. 40–8°. B.p. 168–9°/13 mm. Very spar. sol. H_2O. *Et ester*: $C_{11}H_{19}O_2N$. MW, 197. B.p. 242–3°/749 mm., 113–14°/11–12 mm. D^{20} 0·943.

Shonle, Moment, *J. Am. Chem. Soc.*, 1923, **45**, 248.
Fischer, Flatau, *Ber.*, 1909, **42**, 2984.

1-Propyl-2-isopropylsuccinic Acid (2-*Methylheptane*-3 : 4-*dicarboxylic acid*)

$CH_3·CH_2·CH_2·CH·COOH$
$(CH_3)_2CH·CH·COOH$

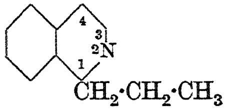
$C_{10}H_{18}O_4$ MW, 202

Cis :
Plates from $CHCl_3$–pet. ether. M.p. 151–2°. $k = 2·95 \times 10^{-4}$ at 25°. HCl at 200° ⟶ part. to *trans* form.
Anhydride : $C_{10}H_{16}O_3$. MW, 184. B.p. 265–75°/742 mm.

Trans :
Cryst. from C_6H_6–pet. ether. M.p. 192–4°. $k = 1·47 \times 10^{-4}$ at 25°. HCl at 200° ⟶ part. to *cis* form.

Bone, Sprankling, *J. Chem. Soc.*, 1900, **77**, 660.

1-Propylisoquinoline



$C_{12}H_{13}N$ MW, 171

B.p. 140–60°/10 mm.
Picrate : m.p. 200–1°.

Späth, Berger, Kuntara, *Ber.*, 1930, **63**, 138.

3-Propylisoquinoline.
Oil with characteristic odour. B.p. 271°. D^{24} 1·0156.
B,HAuCl₄ : yellow needles. M.p. 118° decomp.
B_2,H_2PtCl_6 : yellow needles. Decomp. at 189° without melting.
Picrate : m.p. 161°.

Albahary, *Ber.*, 1896, **29**, 2397.

Propyl isothiocyanate

$CH_3·CH_2·CH_2·N:CS$

C_4H_7NS MW, 101

B.p. 152·7°/743 mm. D_4^{16} 0·9781. n_D^{16} 1·5085.

Delépine, *Ann. chim. phys.*, 1912, **25**, 560.

1-Propylisovaleric Acid.
See 2-Methylhexane-3-carboxylic Acid.

2-Propylkairoline.
See under 2-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Propylmaleic Acid (*Ethylcitraconic acid*, 1-*pentene*-1 : 2-*dicarboxylic acid*)

$CH_3·CH_2·CH_2·C·COOH$
$HC·COOH$

$C_7H_{10}O_4$ MW, 158

Prisms from H_2O, needles from Et_2O–ligroin. M.p. 93–5° (92–4°). Sol. H_2O, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Heat ⟶ propylmaleic anhydride. H_2O at 135–50° ⟶ mainly propylidene-succinic acid. NaHg ⟶ propylsuccinic acid. Ag salt spar. sol. H_2O. Ba salt less sol. hot H_2O than cold.
Anhydride : $C_7H_8O_3$. MW, 140. Oil. B.p. 152–3°/68 mm.

Fittig, Glaser, *Ann.*, 1899, **304**, 184.

Propylmalonic Acid (*Butane*-1 : 1-*dicarboxylic acid*)

$$CH_3·CH_2·CH_2·CH<^{COOH}_{COOH}$$

$C_6H_{10}O_4$ MW, 146

Plates from C_6H_6. M.p. 96°. Sol. to 45·6 parts in 100 parts H_2O at 0°. $k = 1·13 \times 10^{-3}$ at 25°. At 180° ⟶ *n*-valeric acid.
Di-Me ester : $C_8H_{14}O_4$. MW, 174. B.p. 203°/756 mm. D_4^{20} 1·0398. n_D^{20} 1·42155.
Di-Et ester : $C_{10}H_{18}O_4$. MW, 202. B.p. 221° (222–7°/750 mm.), 193·5–194·5°/330 mm. D_{15}^{15} 0·99309, D_{25}^{25} 0·98541.
Diamide : $C_6H_{12}O_2N_2$. MW, 144. Cryst. from H_2O. M.p. 184°. Spar. sol. EtOH, cold H_2O. Insol. Et_2O, $CHCl_3$.
Mononitrile : 1-cyano-*n*-valeric acid, propylcyanoacetic acid. $C_6H_9O_2N$. MW, 127. Oil. B.p. 125–30°/0·2 mm. *Et ester* : $C_8H_{13}O_2N$. MW, 155. B.p. 218–19°/755 mm., 105–10°/15 mm. D^{32} 0·972. *Amide* : 1-cyano-*n*-valeramide, propylcyanoacetamide. $C_6H_{10}ON_2$. MW, 126. Prisms from Et_2O. M.p. 124–124·5° (118°). B.p. 281°. Sol. Et_2O, $CHCl_3$. Mod. sol. hot H_2O. *Anilide* : m.p. 88–9°.
Dinitrile : $C_6H_8N_2$. MW, 108. Oil. B.p. 210°/750 mm. D^{18} 0·9224.
Monohydrazide : $C_6H_{12}O_3N_2$. MW, 160. Cryst. from H_2O. M.p. 139° decomp. *K salt* : m.p. 120°. Very hygroscopic.
Dihydrazide : $C_6H_{14}O_2N_4$. MW, 174. Prisms from EtOH. M.p. 164°. *B,2HCl* : m.p. 180°. *N : N′-Diacetyl* : m.p. 245°.
Dianilide : prisms. M.p. 198°.
*Di-*p*-toluidide* : prisms. M.p. 186°.

Fürth, *Monatsh.*, 1888, **9**, 309.
Bischoff, *Ber.*, 1895, **28**, 2619.
Fischer, Brieger, *Ber.*, 1915, **48**, 1520.
Henry, *Jahresber. Fortschr. Chem.*, 1889, 638.
Vogel, *J. Chem. Soc.*, 1934, 333.
Curtius, Lehmann, *J. prakt. Chem.*, 1930, **125**, 224.
Morton, Davidson, Best, *J. Am. Chem. Soc.*, 1942, **64**, 2239.

Propyl Mercaptan (1-*Mercaptopropane*, *thiopropyl alcohol*)

$CH_3·CH_2·CH_2SH$

C_3H_8S MW, 76

M.p. − 113·3°. B.p. 67°. D_4^0 0·86169, D_4^{25} 0·83572. n_D^{25} 1·4351.
3 : 5-*Dinitrobenzoyl* : cryst. from AcOH.Aq. M.p. 51–2°.
Acid 3-nitrophthalate : cryst. from AcOH.Aq. M.p. 136–7°.

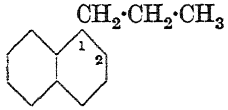
$Hg(S{\cdot}C_3H_7)_2$: leaflets. M.p. 71–2°.

Ellis, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 1674.
Wertheim, *J. Am. Chem. Soc.*, 1929, **51**, 3661.

Propyl *p*-methoxyphenyl Ketone.
See under *p*-Hydroxybutyrophenone.

Propyl *p*-methoxyphenyl sulphide.
See under Thiohydroquinone.

1-Propylnaphthalene



$C_{13}H_{14}$ MW, 170

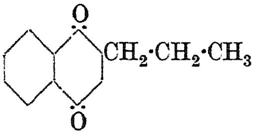
M.p. −13° to −12°. B.p. 272–3°. n_D^{20} 1·5923.
Picrate: yellow needles. M.p. 84–6° decomp.

Morrell, Pickering, Smith, *J. Inst. Petroleum*, 1948, **34**, 677.
Robbin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 158.

2-Propylnaphthalene.
M.p. −3°. B.p. 273–4°. n_D^{20} 1·5872.
Picrate: m.p. 90–2°.

Morrell, Pickering, Smith, *J. Inst. Petroleum*, 1948, **34**, 677.
Robbin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 158.

2-Propyl-1 : 4-naphthoquinone



$C_{13}H_{12}O_2$ MW, 200

Pale yellow needles. M.p. 39–39·5°. Red col. with alc. alkalis.

Fieser, Campbell, Fry, *J. Am. Chem. Soc.*, 1939, **61**, 2206.

Propyl naphthyl Ether.
See under Naphthol.

Propyl naphthyl Ketone.
See Butyronaphthone.

Propylnitramine (N-*Nitropropylamine*, 1-*nitraminopropane*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}NO_2$

$C_3H_8O_2N_2$ MW, 104

Colourless liq. Cryst. on cooling. M.p. − 21°. B.p. 128–9°/40 mm. D^{15} 1·1046. Misc. with EtOH, Et_2O. Spar. sol. H_2O ⟶ acid sol.

Umbgrove, Franchimont, *Rec. trav. chim.*, 1898, **17**, 272.

Propyl nitrate

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}NO_2$

$C_3H_7O_3N$ MW, 105

B.p. 110·5°. D_4^{20} 1·0548. n_D^{20} 1·3979.

Cowley, Partington, *J. Chem. Soc.*, 1933, 1252.
Wallach, Schulze, *Ber.*, 1881, **14**, 421.

Propyl nitrite

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}NO$

$C_3H_7O_2N$ MW, 89

B.p. 46–8° (57°). D_4^{20} 0·8861. n_D^{20} 1·3604. H (+ Ni) at 130° ⟶ propylamine; at 200° ⟶ a mixture of mono-, di- and tri-propylamines.

Cowley, Partington, *J. Chem. Soc.*, 1933, 1253.

Propyl pentadecyl Ketone (*Nonadecanone*-4)

$CH_3{\cdot}[CH_2]_{14}{\cdot}CO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_{19}H_{38}O$ MW, 282

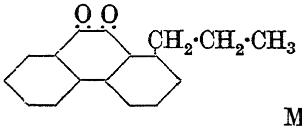
M.p. 50·5°. B.p. 211°/11 mm., part. decomp. Spar. sol. EtOH.
Oxime: exists in two forms. (i) M.p. 25·5–26·5°. (ii) M.p. 43·5–44·5°.

Bertrand, *Bull. soc. chim.*, 1896, **15**, 766.
Furukawa, *Chem. Abstracts*, 1933, **27**, 2131.

2-Propyl-2-pentene.
See 4-Methyl-3-heptene.

4-Propyl-2-pentene.
See 4-Methyl-2-heptene.

1-Propylphenanthraquinone

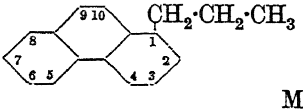


$C_{17}H_{14}O_2$ MW, 250

Orange plates from EtOH. M.p. 139–40°.
Quinoxaline deriv.: pale yellow needles from AcOH. M.p. 144–5°.

Haworth, Mavin, Sheldrick, *J. Chem. Soc.*, 1934, 460.

1-Propylphenanthrene



$C_{17}H_{16}$ MW, 220

Plates from MeOH. M.p. 34–5°.
Picrate: yellow needles from MeOH. M.p. 100–1°.

Haworth, Mavin, Sheldrick, *J. Chem. Soc.*, 1934, 460.

2-Propylphenanthrene.
Leaflets from EtOH. M.p. 35–6°. B.p. 170°/0·2 mm.
Picrate: yellow needles from EtOH. M.p. 91–2°.

Wilds, *J. Am. Chem. Soc.*, 1942, **64**, 1421.

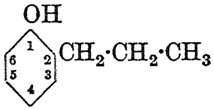
9-Propylphenanthrene.
Plates from EtOH. M.p. 59°. B.p. 265–70°/22 mm.

Picrate: yellow needles from EtOH. M.p. 99°.

Bachman, Hoaglin, *J. Am. Chem. Soc.*, 1941, **63**, 621.

Propylphenetole.
See under Propylphenol.

o-Propylphenol (2-*Hydroxy*-1-*propylbenzene*)



$C_9H_{12}O$ MW, 136

Oil. B.p. 220–220·5°. D_{15}^{15} 1·000.
Me ether: 2-methoxy-1-propylbenzene, *o*-propylanisole. $C_{10}H_{14}O$. MW, 150. B.p. 207–9°/757·7 mm. D^0 0·96944.
Et ether: 2-ethoxy-1-propylbenzene, *o*-propylphenetole. B.p. 213°/754 mm., 99–100°/16 mm. D_4^{26} 0·92396. n_D^{26} 1·494.
Propionyl: b.p. 245°.
Phenylurethane: needles from formic acid. M.p. 111°.

Claisen, *Ann.*, 1919, **418**, 87.
Farinholt, Harden, Twiss, *J. Am. Chem. Soc.*, 1933, **55**, 3386.

m-Propylphenol (3-*Hydroxy*-1-*propylbenzene*).
B.p. 228°. Spar. sol. H_2O. $FeCl_3 \longrightarrow$ green col. in EtOH, bluish col. in H_2O.
Me ether: 3-methoxy-1-propylbenzene, *m*-propylanisole. B.p. 212–13°.
Et ether: 3-ethoxy-1-propylbenzene, *m*-propylphenetole. B.p. 220–4°/753 mm., 109–10°/15 mm. D_4^{20} 0·94558. n_D^{20} 1·5025.

Henrard, *Chem. Zentr.*, 1907, II, 1512.
Ciamician, Silber, *Ber.*, 1890, **23**, 1162.

p-Propylphenol (4-*Hydroxy*-1-*propylbenzene*).
Cryst. M.p. 21–2°. B.p. 230–2°, 120°/19 mm. D_4^0 1·089. n_D^{25} 1·5379.
Me ether: 4-methoxy-1-propylbenzene, *p*-propylanisole, dihydroanethole. B.p. 215–16°, 86·5°/10 mm. D_4^{20} 0·94718. n_D^{20} 1·5045.
Et ether: 4-ethoxy-1-propylbenzene, *p*-propylphenetole. B.p. 223–30°, 108–10°/13 mm. D_4^{15} 0·94.
Acetyl: b.p. 245–6°/745 mm. D^0 1·02904.
Propionyl: b.p. 254–6°.
Benzoyl: m.p. 37–8°.
Salicyloyl: m.p. 57°.
Phenylurethane: cryst. from EtOH. M.p. 128·5–129°.

Farinholt, Harden, Twiss, *J. Am. Chem. Soc.*, 1933, **55**, 3386.
Baranger, *Bull. soc. chim.*, 1931, **49**, 1213.
Albright, *J. Am. Chem. Soc.*, 1914, **36**, 2197.
Clemmensen, *Ber.*, 1914, **47**, 53.
Ipatjew, *Ber.*, 1913, **46**, 3590

1-Propyl-1-phenyl-1-butylene.
See 4-Phenyl-3-heptene.

Propylphenylcarbinol (1-*Phenyl*-n-*butyl alcohol*, α-*hydroxybutylbenzene*, α-*propylbenzyl alcohol*)

$CH_3 \cdot CH_2 \cdot CH_2 \cdot CH(OH) \cdot C_6H_5$

$C_{10}H_{14}O$ MW, 150

d-.
M.p. 49°. B.p. 115°/14 mm. $[\alpha]_{5461}$ + 52·2° in C_6H_6.
Acetyl: b.p. 125°/16 mm. $n_D^{20·5}$ 1·4889.
Acid phthalate: m.p. 53–4°. $[\alpha]_{5461}$ + 11·5° in Et_2O.

l-.
M.p. 48–9°. $[\alpha]_{5461}$ − 53·5° in C_6H_6.
Acid phthalate: m.p. 52–3°. $[\alpha]_{5461}$ − 11·0° in Et_2O.

dl-.
Oil with aromatic odour. B.p. 168–70°/100 mm., 113–15°/10 mm. $D_4^{13·7}$ 0·9861. $n_D^{13·7}$ 1·51914.
Acetyl: b.p. 117–18°/8 mm.
Benzoyl: b.p. 145–6°/1 mm. D_{25}^{25} 1·0590. n_D^{25} 1·5446.
Acid phthalate: m.p. 90–1°.

Grignard, *Ann. chim. phys.*, 1901, **24**, 466.
Kenyon, Partridge, *J. Chem. Soc.*, 1936, 128.
Klages, *Ber.*, 1904, **37**, 2312.

Propyl-phenylethyl-carbinol.
See 1-Phenylhexanol-3 *and* 2-Phenylhexanol-3.

Propyl phenylethyl Ketone.
See 1-Phenylhexanone-3 *and* 2-Phenylhexanone-3.

Propylphenylglycollic Acid.
See 1-Hydroxy-1-phenylbutyric Acid.

3-Propyl-3-phenylhexane.
See 4-Ethyl-4-phenylheptane.

Propyl phenyl Ketone.
See Butyrophenone.

Propylphenylpropylene.
See 1-Phenyl-2-hexene.

Propyl phenyl sulphone

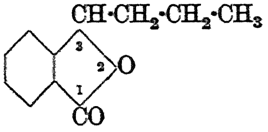
$C_6H_5 \cdot SO_2 \cdot CH_2 \cdot CH_2 \cdot CH_3$

$C_9H_{12}O_2S$ MW, 184

Plates from ligroin. M.p. 46° (44°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. boiling H_2O.

Baldwin, Robinson, *J. Chem. Soc.*, 1932, 1448.

3-Propylphthalide



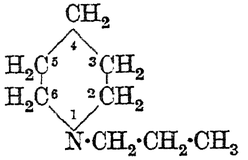
$C_{11}H_{12}O_2$ MW, 176

Oil with odour of celery. M.p. 20°. B.p. 293–7°/735 mm. decomp., 243–7°/220 mm., 150°/14 mm. D_{15}^{15} 1·1073. $n_D^{17·5}$ 1·5327. Volatile in steam. NaOH fusion ⟶ butyric and benzoic acids.

Tasman, *Rec. trav. chim.*, 1927, **46**, 653.

N-Propylphthalimide.
See under Phthalimide.

N-Propylpiperidine (*Propylpiperidylamine*)



$C_8H_{17}N$ MW, 127

B.p. 152°. D_{20}^{20} 0·8231. n_D^{20} 1·4446.
B,HCl: m.p. 212–13°. Hygroscopic.
Chloroplatinate: m.p. 179°.
Picrate: yellow needles. M.p. 121° (108°).
Methiodide: m.p. 181–2°.
Ethiodide: m.p. 276·5°.

v. Braun, *Ber.*, 1909, **42**, 2048.
Auerbach, Wolffenstein, *Ber.*, 1899, **32**, 2511.
Ladenburg, *Ber.*, 1881, **14**, 1348.

2-Propylpiperidine.
See Coniine.

3-Propylpiperidine.

d-.
$[\alpha]_D^{15}$ + 5·9°.
B,HCl: cryst. from Me_2CO. M.p. 147°. Very sol. H_2O, EtOH.
l-*Tartrate*: cryst. from H_2O. M.p. 161°.

l-.
B.p. 174°/752·5 mm. D_4^{19} 0·8517. $[\alpha]_D^{16}$ − 6·6°.
B,HCl: needles from Me_2CO. M.p. 147°. Very sol. H_2O, EtOH.
d-*Tartrate*: cryst. from H_2O. M.p. 161°.

dl-.
Liq. with odour resembling coniine. B.p. 174°/758 mm. D_4^{26} 0·8475. Sol. in about 80 parts H_2O at ord. temp. Turns brown in air.
B,HCl: prisms or needles from H_2O. M.p. 127–9°. Sol. H_2O, EtOH. Insol. Et_2O.
B,HAuCl$_4$: lemon-yellow needles from H_2O. M.p. 95–8°.
B$_2$,H$_2$PtCl$_6$: orange-yellow needles. M.p. 94° (slow heat.), 134° (rapid heat.).
Picrate: yellow needles. M.p. 121·5°.

Granger, *Ber.*, 1895, **28**, 1203; 1897, **30**, 1060.

4-Propylpiperidine.
Liq. with odour resembling coniine. B.p. 178–80°.

Ahrens, *Ber.*, 1905, **38**, 159.

Propylpiperidylamine.
See N-Propylpiperidine.

Propylpropenylcarbinol.
See 2-Heptenol-4.

Propylpropenylethylene.
See 2 : 4-Octadiene.

Propyl propenyl Ketone.
See 2-Heptenone-4.

Propylpropiolic Acid (1-*Pentyne*-1-*carboxylic acid*)

$CH_3·CH_2·CH_2·C{:}C·COOH$

$C_6H_8O_2$ MW, 112

Feathery cryst. M.p. 27°. B.p. 126–7°/24 mm., 119–21°/16 mm. Sol. EtOH, Et_2O, ligroin. Somewhat difficultly sol. H_2O. Dist. at atmospheric press. ⟶ propylacetylene. Hot KOH.Aq. ⟶ methyl propyl ketone. Hot alc. KOH ⟶ butyrylacetic acid.
Me ester: $C_7H_{10}O_2$. MW, 126. B.p. 80–2°/23 mm. D^0 0·9648.
Et ester: $C_8H_{12}O_2$. MW, 140. B.p. 93–4°/24 mm. D^0 0·9468.
Isoamyl ester: $C_{11}H_{18}O_2$. MW, 182. B.p. 127–8°/22 mm. D^0 0·9207.

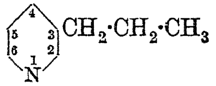
Moureu, Delange, *Bull. soc. chim.*, 1903, **29**, 652.

Propylpropionylcarbinol.
See 4-Heptanolone-3.

2-Propylpropylene.
See 2-Methyl-1-pentene.

2-Propylpyridine.
See Conyrine.

3-Propylpyridine



$C_8H_{11}N$ MW, 121

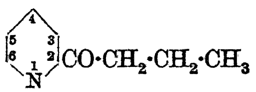
B.p. 170°. Ox. ⟶ nicotinic acid.

Cahours, Étard, *Compt. rend.*, 1881, **92**, 1082.

4-Propylpyridine.
B.p. 184–6°. D^{15} 0·9381. Sol. EtOH, Et_2O. Spar. sol. H_2O.
B,HCl: needles from EtOH.Aq. M.p. 215°.
B,HAuCl$_4$: yellow cryst. M.p. 113–15°.
B$_2$,H$_2$PtCl$_6$: brownish-yellow cryst. M.p. 204°.
Picrate: needles from EtOH. M.p. 153°.

Koenigs, Jaeschke, *Ber.*, 1921, **54**, 1355.
Arens, Wibaut, *Rec. trav. chim.*, 1942, **61**, 59.

Propyl 2-pyridyl Ketone (2-*Butyrylpyridine*)



$C_9H_{11}ON$ MW, 149

Oil with characteristic odour. B.p. 217–18° (216–20°). Sol. acids.

Oxime: needles from pet. ether. M.p. 48°.
Benzoyl: yellow leaflets from C_6H_6. M.p. 56–7°.
Phenylhydrazone: yellowish needles from EtOH. M.p. 82°. Unstable.
p-*Sulphophenylhydrazone*: yellow needles. M.p. 251°.
Picrate: yellow needles from H_2O. M.p. 75°.
Methiodide: yellow needles from EtOH–Et_2O. M.p. 79°.

Engler, Majmon, *Ber.*, 1891, **24**, 2536.
Pinner, *Ber.*, 1901, **34**, 4243.

Propyl 3-pyridyl Ketone (*3-Butyrylpyridine*).

Liq. with odour resembling coniine. B.p. 246–52°. Sol. EtOH, Et_2O, acids.
Phenylhydrazone: yellow cryst. M.p. 182° (129–30°).
Semicarbazone: m.p. 169–70°.
Ethiodide: yellow cryst. M.p. 192°.

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2477.
Engler, *Ber.*, 1891, **24**, 2541.

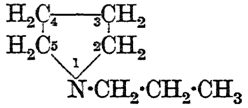
Propyl 4-pyridyl Ketone (*4-Butyrylpyridine*).

B.p. 229–31°.
Picrate: yellow needles from H_2O. M.p. 96°.

Pinner, *Ber.*, 1901, **34**, 4252.

Propylpyrogallol dimethyl Ether.
See Picamar.

N-Propylpyrrolidine



$C_7H_{15}N$ MW, 113

B.p. 130°. D_4^{20} 0·8171. n_D^{20} 1·4389. Sol. H_2O.
B_2,H_2PtCl_6: red cryst. Decomp. at 184–90°.
Picrate: yellow plates. M.p. 105° (101°).

v. Braun, *Ber.*, 1911, **44**, 1254.
Jurjew, Schenjan, *Chem. Zentr.*, 1936, I, 4293.
Coleman, Nichols, Martens, *Organic Syntheses*, 1945, XXV, 14.

2-Propylpyrrolidine.

Oil with odour resembling piperidine. B.p. 145–50°/765 mm. Sol. H_2O with alk. reaction.
Chloroaurate: yellow needles or leaflets. M.p. 120°.
Chloroplatinate: m.p. anhyd. 135°.
Picrate: m.p. 104–104·5°.
N-*Me*: $C_8H_{17}N$. MW, 127. B.p. 146–7°. D^{15} 0·815. *$B,HAuCl_4$*: yellow leaflets. M.p. 76°. *B_2,H_2PtCl_6*: orange leaflets. M.p. 145–6°. *Picrate*: yellow needles from EtOH. M.p. 124°.

N-*Benzenesulphonyl*: needles from 80% EtOH. M.p. 66–67·5°.

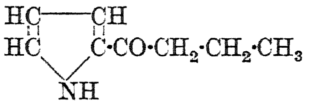
Hess, *Ber.*, 1913, **46**, 4110.
Löffler, *Ber.*, 1910, **43**, 2039.
Gabriel, *Ber.*, 1909, **42**, 1264.

3-Propylpyrrolidine.

Oil with odour resembling piperidine. B.p. 158–60°/746 mm. D_4^{20} 0·8450. n_D^{20} 1·4469. Fumes in air. Absorbs H_2O and CO_2 from the air.

Longinow, *Chem. Zentr.*, 1915, I, 982.

Propyl 2-pyrryl Ketone (*2-Butyrylpyrrole*)

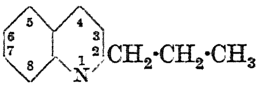


$C_8H_{11}ON$ MW, 137

Cryst. from H_2O. M.p. 48·5°. B.p. 235°. Sol. EtOH, Et_2O, C_6H_6, pet. ether, KOH.Aq.
Phenylhydrazone: yellow cryst. from pet. ether. M.p. 80·5°.
Semicarbazone: needles from H_2O. M.p. 131°.

Tschelinzeff, Terentjeff, *Ber.*, 1914, **47**, 2650.
Oddo, *Ber.*, 1910, **43**, 1016.

2-Propylquinoline



$C_{12}H_{13}N$ MW, 171

Pale yellowish-green oil. B.p. 142–5°/13 mm., 130–1°/10 mm. D_4^{17} 1·038. n_D^{23} 1·5886.
Chloromercurate: m.p. 112°.
Methiodide: m.p. 184°.
Picrate: yellow needles or leaflets from EtOH. M.p. 163–4°.
Methopicrate: yellow needles. M.p. 118°.

Freund, Kessler, *J. prakt. Chem.*, 1918, **98**, 233.
Meisenheimer, Schütze, *Ber.*, 1923, **56**, 1353.
Delaby, Hiron, *Compt. rend.*, 1930, **191**, 845.

4-Propylquinoline.

Liq. with odour resembling quinoline. B.p. 159°/16 mm.
B,HCl: m.p. 156–7°.
$B,HBr,CdBr_2$: cryst. from EtOH. M.p. 148°.
$B_2,2HCl,HgCl_2$: cryst. from EtOH. M.p. 148°.
B_2,H_2PtCl_6: cryst. from conc. HCl. M.p. 198° decomp.
Picrate: yellow needles from EtOH. M.p. 204°.

Koenigs, *Ber.*, 1898, **31**, 2376.
Blaise, Maire, *Bull. soc. chim.*, 1908, **3**, 667.

8-Propylquinoline.

Pale yellow liq. B.p. 142°/15 mm.

B_2,H_2PtCl_6: m.p. 196°. Very spar. sol. hot H_2O.

Picrate: yellowish-red needles from EtOH. M.p. 142°.

Methiodide: m.p. 136°.

v. Braun, Heider, Wyczatkowska, *Ber.*, 1918, **51**, 1215.

Propylresorcinol.

See 2 : 4- *and* 2 : 6-Dihydroxypropylbenzene, *and* Divarinol.

Propylsuccinic Acid (*Pentane*-1 : 2-*dicarboxylic acid*)

$CH_3·CH_2·CH_2·CH·COOH$
$CH_2·COOH$

$C_7H_{12}O_4$ MW, 160

d-.

M.p. 93·9°. $[\alpha]_D + 9·6°$.

dl-.

Cryst. from H_2O or C_6H_6. M.p. 100·5° (92–3°). 2·83 parts sol. 100 parts cold $CHCl_3$. *k* (first) = $8·9 \times 10^{-5}$ at 25°; *k* (second) = $1·2 \times 10^{-6}$ at 100°.

Di-Me ester: $C_9H_{16}O_4$. MW, 188. B.p. 112°/15 mm., 107°/11 mm.

Di-Et ester: $C_{11}H_{20}O_4$. MW, 216. B.p. 132–4°/25 mm.

Diamide: $C_7H_{14}O_2N_2$. MW, 158. Needles from EtOH. M.p. 234–5°. Spar. sol. EtOH.

Anhydride: $C_7H_{10}O_3$. MW, 142. Viscous liq. B.p. 145–55°/20 mm.

Dihydrazide: powder from EtOH. M.p. 176°.

Locquin, *Bull. soc. chim.*, 1909, **5**, 1073.
Fittig, Glaser, *Ann.*, 1899, **304**, 188.
Timmermans, van der Haegen, *Bull. soc. chim. Belg.*, 1933, **42**, 448.
Scheibler, Schmidt, *Ber.*, 1921, **54**, 153.

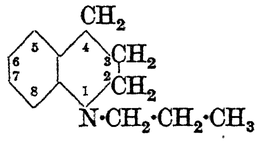
β-Propylsulphenylalanine.

See Dihydroalliin.

2-Propyl-1 : 4 : 5 : 6-tetrahydropyridine.

See γ-Coniceine.

***N*-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline**



$C_{12}H_{17}N$ MW, 175

Colourless liq. B.p. 146°/16 mm. Gradually turns reddish-brown.

B,HCl: m.p. 162°.

B,HBr: m.p. 177°. Somewhat difficultly sol. cold H_2O.

B,HI: needles. M.p. 178°. Insol. cold H_2O.

Picrate: m.p. 73°.

Methiodide: kairoline propiodide. Plates from EtOH–Et_2O. M.p. 135°.

v. Braun, *Ber.*, 1909, **42**, 2222.

2-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Oil with faint violet fluor. B.p. 258°/746 mm., 152°/20 mm., 140–1°/10 mm. D^{17} 0·959. $n_D^{14·5}$ 1·5673.

B,HCl: m.p. 221°. Spar. sol. EtOH, hot H_2O. Sublimes.

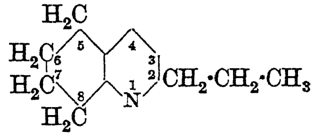
Picrate: orange plates from EtOH or Et_2O. M.p. 125°.

N-*Me*: 2-propylkairoline. $C_{13}H_{19}N$. MW, 189. B.p. 272–6°, 151–7°/20 mm., 144·5°/10 mm. Volatile in steam. *Picrate*: yellow leaflets from EtOH. M.p. 123° (120°). *Methiodide*: melts between 180–200° ⟶ its components.

N-*Benzoyl*: leaflets from EtOH or Et_2O. M.p. 102° (97°).

v. Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 382.
Tröger, Ungar, *J. prakt. Chem.*, 1926, **112**, 254.
Meisenheimer, Schütze, *Ber.*, 1923, **56**, 1357.

2-Propyl-5 : 6 : 7 : 8-tetrahydroquinoline



$C_{12}H_{17}N$ MW, 175

B.p. 130–2°/11 mm.

B_2,H_2PtCl_6: decomp. at 62°.

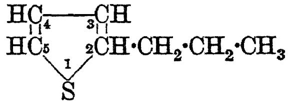
Picrate: yellow cryst. from EtOH. M.p. 119°.

v. Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 382.

1-Propyltetramethylene Glycol.

See Heptandiol-1 : 4.

2-Propylthiophene



$C_7H_{10}S$ MW, 126

B.p. 157–60°. D_{20}^{20} 0·9700, D_4^{20} 0·9683. n_D^{20} 1·5048.

Scheibler, Schmidt, *Ber.*, 1921, **54**, 149.
Steinkopf, Schubart, *Ann.*, 1921, **424**, 21.

3-Propylthiophene.

B.p. 160–2°. D_{20}^{20} 0·9733, D_4^{20} 0·9716. n_D^{20} 1·5057. Isatin + H_2SO_4 ⟶ blue col.

Scheibler, Schmidt, *Ber.*, 1921, **54**, 153.

Propylthiourea

$H_2N·CS·NH·CH_2·CH_2·CH_3$
3 2 1

$C_4H_{10}N_2S$ MW, 118

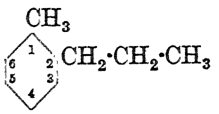
Needles from EtOH. M.p. 110°. Sol. EtOH. Mod. sol. H_2O.

3-N-*Me*: plates from EtOH.Aq. M.p. 79°. Sol. EtOH, Me_2CO, $CHCl_3$. Mod. sol. H_2O. Insol. ligroin.

3-N-*Et*: plates from EtOH. M.p. 52°.

Hecht, *Ber.*, 1890, **23**, 283.

o-Propyltoluene (*2-Methylpropylbenzene*)



$C_{10}H_{14}$ MW, 134

B.p. 184°, 65–8°/14 mm. D_4^{19} 0·8747. n_D^{20} 1·4995.

Claus, Hansen, *Ber.*, 1880, **13**, 897.
Auwers, *Ann.*, 1919, **419**, 111.
Kuhn, Deutsch, *Ber.*, 1932, **65**, 48.

m-Propyltoluene (*3-Methylpropylbenzene*).

B.p. 181·5–182·5°. D_4^{17} 0·8648, D_4^{20} 0·862. n_D^{20} 1·4951.

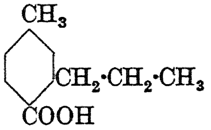
Auwers, *Ann.*, 1919, **419**, 111.
Claus, Stüsser, *Ber.*, 1880, **13**, 899.

p-Propyltoluene (*4-Methylpropylbenzene*).

B.p. 183–4°. $D_4^{15·4}$ 0·8642. n_D^{22} 1·4823.

Bayrac, *Bull. soc. chim.*, 1895, **13**, 894.
Auwers, *Ann.*, 1919, **419**, 112.
Ipat'ev, Orlov, Petrov, *Chem. Abstracts*, 1931, **25**, 4540.

3-Propyl-p-toluic Acid (*4-Methyl-2-propylbenzoic acid*)

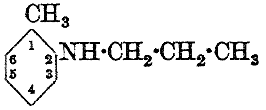


$C_{11}H_{14}O_2$ MW, 178

Needles from H_2O. M.p. 75–6°. Volatile in steam.

Claus, *J. prakt. Chem.*, 1892, **46**, 495.

N-Propyl-o-toluidine



$C_{10}H_{15}N$ MW, 149

Oil. B.p. 230°.

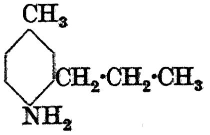
Bischoff, Mintz, *Ber.*, 1892, **25**, 2319.

N-Propyl-p-toluidine.

Oil with odour of caraway. B.p. 235°/761 mm. D^{20} 0·9243, D^{35} 0·9172. n_D 1·5367.
B,HCl: needles. M.p. 150–1°.
B_2,$(COOH)_2$: cryst. M.p. 116–17°.
B,$(COOH)_2$: m.p. 172–3° decomp.

Hori, Morley, *J. Chem. Soc.*, 1891, **59**, 35.
Bischoff, Mintz, *Ber.*, 1892, **25**, 2321.

3-Propyl-p-toluidine (*4-Methyl-2-propylaniline*)



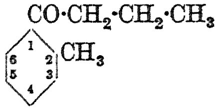
$C_{10}H_{15}N$ MW, 149

B.p. 98–9°/13 mm. D_4^{22} 0·9666.
B,HCl: cryst. from H_2O. M.p. 195°.
N-*Benzoyl*: m.p. 174·5°.
Picrate: cryst. from EtOH. M.p. 201°.

v. Braun, Bayer, Blessing, *Ber.*, 1924, **57**, 402.

Propyl tolyl Ether.
See under Cresol.

Propyl o-tolyl Ketone (*2-Methylbutyrophenone*)



$C_{11}H_{14}O$ MW, 162

B.p. 238·5°/758 mm. D_4^0 0·9936.
Semicarbazone: m.p. 176°.

Senderens, *Bull. soc. chim.*, 1911, **9**, 949.

Propyl m-tolyl Ketone (*3-Methylbutyrophenone*).

B.p. 247°/758 mm. D_4^0 0·9882.
Semicarbazone: m.p. 152°.

Senderens, *Bull. soc. chim.*, 1911, **9**, 949.

Propyl p-tolyl Ketone (*4-Methylbutyrophenone*).

B.p. 251·5°/758 mm. D_{20}^{20} 0·9660. n_D^{20} 1·5232. Sol. EtOH, Et_2O.
Semicarbazone: m.p. 190° (232°).
Phenylhydrazone: m.p. 73°.

Senderens, *Bull. soc. chim.*, 1911, **9**, 949.
Willgerodt, Hambrecht, *J. prakt. Chem.*, 1910, **81**, 78.
Blaise, *Compt. rend.*, 1901, **133**, 1217.

1-Propyltricarballylic Acid.
See Hexane-1 : 2 : 3-tricarboxylic Acid.

Propylurea

$H_2\underset{3}{N}{\cdot}\underset{2}{CO}{\cdot}\underset{1}{NH}{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$

$C_4H_{10}ON_2$ MW, 102

Prisms from EtOH. M.p. 110°. Sol. EtOH. Mod. sol. H_2O.
3-*Acetyl*: plates from CS_2. M.p. 115°. Mod. sol. H_2O, EtOH, Et_2O, $CHCl_3$, C_6H_6.

Hecht, *Ber.*, 1890, **23**, 283.
Mauguin, *Ann. chim. phys.*, 1911, **22**, 343.

Propylurethane (*Ethyl propylaminoformate, ethyl propylcarbamate*)

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CO{\cdot}OC_2H_5$

$C_6H_{13}O_2N$ MW, 131

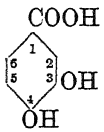
B.p. 191·5–192·5°/758 mm.

Schreiner, *J. prakt. Chem.*, 1880, **21**, 125.
Nirdlinger, Acree, *Am. Chem. J.*, 1910, **43**, 378.

1-Propylvaleraldehyde.
See Dipropylacetaldehyde.

1-Propylvaleric Acid.
See Dipropylacetic Acid.

2-Propylvaleric Acid.
See 2-Ethylcaproic Acid.
3-Propylvaleric Acid.
See 3-Methyl-*n*-heptylic Acid.
Propylveratrol.
See under Cœrulignol *and under* 2 : 3-Dihydroxy-1-propylbenzene.
Propylvinylacetylene.
See 1-Hepten-3-yne.
Propylvinylcarbinol.
See 1-Hexenol-3.
Propyl vinyl Ketone.
See 1-Hexenone-3.
Propyne.
See Allylene.
Propyne-1 : 3-dicarboxylic Acid.
See Glutinic Acid.
Prothebenine.
See under Thebenine.
Protocatechuic Acid (3 : 4-*Dihydroxybenzoic acid, catechol*-4-*carboxylic acid*)

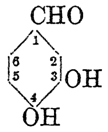


$C_7H_6O_4$ MW, 154

Needles + $1H_2O$ from H_2O. M.p. 199°. Sol. EtOH. Mod. sol. H_2O. Insol. C_6H_6. $k = 3{\cdot}3 \times 10^{-5}$ at 25°. Aq. sol. with $FeCl_3$ ⟶ green col. ⟶ dark red with $NaHCO_3$.
3-*Me ether* : *see* Vanillic Acid.
4-*Me ether* : *see* Isovanillic Acid.
Di-Me ether : *see* Veratric Acid.
Di-Et ether : 3 : 4-diethoxybenzoic acid. $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH. M.p. 165–6°. $k = 3{\cdot}4 \times 10^{-5}$ at 25°. *Et ester* : $C_{13}H_{18}O_4$. MW, 238. Cryst. from EtOH. M.p. 56–7°.
Me ester : $C_8H_8O_4$. MW, 168. Needles from H_2O. M.p. 134·5°. Sol. EtOH. Spar. sol. H_2O. Has antimicrobic properties. 3-*Benzoyl* : m.p. 153·5–155°. 3-*Acetyl*-4-*benzoyl* : cryst. from MeOH. M.p. 102–3°. 4-*Acetyl*-3-*benzoyl* : prisms. M.p. 54–5°.
Et ester : $C_9H_{10}O_4$. MW, 182. Prisms from H_2O. M.p. 133–4°. Sol. EtOH.
Phenyl ester : $C_{13}H_{10}O_4$. MW, 230. Cryst. from EtOH.Aq. M.p. 189°. $FeCl_3$ ⟶ green col.
Amide : $C_7H_7O_3N$. MW, 153. Cryst. from H_2O. M.p. 212°.
Nitrile : $C_7H_5O_2N$. MW, 135. Needles from H_2O. M.p. 156°. Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6, ligroin, xylene. *Diacetyl* : needles from EtOH. M.p. 87°. *Dibenzoyl* : cryst. from EtOH. M.p. 131°.
Anilide : prisms from EtOH. M.p. 166–7°.
3-*Acetyl* : prisms from H_2O. M.p. 202–3°. 4-*Benzoyl* : cryst. from C_6H_6. M.p. 154–5°.
Diacetyl : cryst. from H_2O. M.p. 157–8°.
3-*Benzoyl* : microneedles. M.p. 225–7°.

Hoesch, Zarzecki, *Ber.*, 1917, **50**, 462.
Fischer, Bergmann, Lipschitz, *Ber.*, 1918, **51**, 45.
Schmidt, B.P. 145,081, (*Chem. Abstracts*, 1920, **14**, 3089); D.R.P., 278,778, (*Chem. Zentr.*, 1914, II, 1080).
Ono, Imoto, *J. Chem. Soc. Japan*, 1935, **56**, 715.
Pratt, Perkins, *J. Am. Chem. Soc.*, 1918, **40**, 224.
Miller, *Ann.*, 1883, **220**, 116.
Pearl, *J. Am. Chem. Soc.*, 1946, **68**, 2180.

Protocatechuic Aldehyde (3 : 4-*Dihydroxybenzaldehyde*)



$C_7H_6O_3$ MW, 138

Cryst. from toluene. M.p. 153°.
Oxime : m.p. 157° decomp.
Phenylhydrazone : two forms. (i) M.p. 175–6° decomp. (ii) M.p. 121–8°.
Semicarbazone : decomp. at 230°.
Azine : cryst. from EtOH.Aq. Decomp. about 245°.
2 : 4-*Dinitrophenylhydrazone* : dark red microcryst. from MeOH. M.p. 275° decomp.
3-*Me ether* : *see* Vanillin.
4-*Me ether* : *see* Isovanillin.
Di-Me ether : *see* Veratric Aldehyde.
3-*Et ether* : *see* Bourbonal.
Di-Et ether : $C_{11}H_{14}O_3$. MW, 194. Colourless oil. B.p. 278–80°.
3-*Propyl ether* : $C_{10}H_{12}O_3$. MW, 180. Needles from H_2O. M.p. 82°.
3-*Isobutyl ether* : $C_{11}H_{14}O_3$. MW, 194. Needles from EtOH.Aq. M.p. 94°.
3-*Benzyl ether* : m.p. 113–14°.
4-*Benzyl ether* : plates from EtOH. M.p. 122°.
Methylene ether : *see* Piperonal.
Ethylene ether : $C_9H_8O_3$. MW, 164. Needles from ligroin. M.p. 51·5°. B.p. 299°. *Oxime* : cryst. from EtOH. M.p. 75–75·5°. *Phenylhydrazone* : cryst. from EtOH. M.p. 107–8°. *Azine* : yellow needles from AcOH. M.p. 190–1°.
Carbonate : m.p. 124°. B.p. 289°, 162°/13 mm. Mod. sol. EtOH, Me_2CO, $CHCl_3$, AcOH. Spar. sol. Et_2O, CCl_4.
3-*Acetyl* : plates from C_6H_6. M.p. 109–10°. p-*Nitrophenylhydrazone* : red needles from EtOH. M.p. 195°. 4-*Benzoyl* : needles from EtOH. M.p. 109°. *Phenylhydrazone of* 3-*acetyl*-4-*benzoyl* : needles from EtOH. M.p. 158°.
Diacetyl : cryst. from EtOH. M.p. 54°. *Phenylhydrazone* : needles from EtOH. M.p. 135°. *Semicarbazone* : needles from EtOH. M.p. 200–2° decomp.
3-*Benzoyl* : needles from EtOH. M.p. 136–7°. *Phenylhydrazone* : cryst. from EtOH. M.p.

192°. 4-*Acetyl*: cryst. from EtOH. M.p. 68°. *Phenylhydrazone of 4-acetyl-3-benzoyl*: yellow plates from EtOH. M.p. 166°.

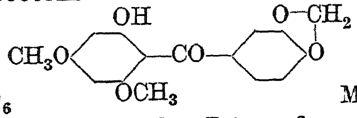
Dibenzoyl: *phenylhydrazone*, yellow plates. M.p. 167°.

Di-carbomethoxyl: m.p. 99–100°. p-*Nitrophenylhydrazone*: m.p. 187–9°.

Gattermann, *Ann.*, 1907, **357**, 374.
Boehringer, D.R.P., 269,544, (*Chem. Zentr.*, 1914, I, 591).
Schmidt, D.R.P., 295,337, (*Chem. Zentr.*, 1917, I, 41).
Hoesch, Zarzecki, *Ber.*, 1917, **50**, 465.
Tiemann, Koppe, *Ber.*, 1881, **14**, 2015.
Pacsu, v. Vargha, *Ber.*, 1926, **59**, 2818.
Fröschl, Bomberg, *Monatsh.*, 1927, **48**, 571.
Roberts, B.P. 417,072, (*Chem. Abstracts*, 1935, **57**, 1099).
Vanillin Fabrik., D.R.P., 591,888, (*Chem. Zentr.*, 1934, I, 2491).
Buck, Zimmermann, *Organic Syntheses*, 1938, XVIII, 75.

Protocetraric Acid.
See Obtusatic Acid.

Protocotoin



$C_{16}H_{14}O_6$ MW, 302

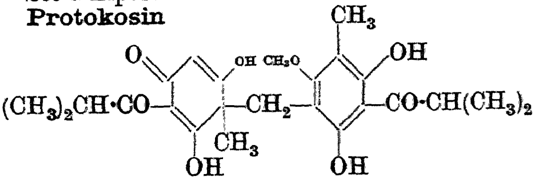
Occurs in Coto bark. Prisms from MeOH. M.p. 141–2°. Sol. Et_2O, $CHCl_3$, AcOH, C_6H_6. Insol. H_2O. Sol. alkalis with yellow col. 65% HNO_3 ⟶ bluish-green col. turning red on warming. $FeCl_3$ in EtOH.Aq. ⟶ reddish-brown col. H_2SO_4 ⟶ orange col.

Me ether: oxyleukotin, methylprotocotoin. $C_{17}H_{16}O_6$. MW, 316. Prisms from EtOH. M.p. 134–5°. Sol. EtOH, AcOH. Mod. sol. Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O. 65% HNO_3 ⟶ green col. turning reddish-brown on warming. H_2SO_4 ⟶ orange col. No. col. with $FeCl_3$ in EtOH.Aq. *Phenylhydrazone*: prisms from EtOH. M.p. 211°. *Acetyl*: cryst. from EtOH. M.p. 103°.

Späth, Bretschneider, *Monatsh.*, 1928, **49**, 429.
Houben, Fischer, *J. prakt. Chem.*, 1929, **123**, 89.
Ciamician, Silber, *Ber.*, 1893, **26**, 779; 1891, **24**, 2984.

Protogen.
See α-Lipoic Acid.

Protokosin



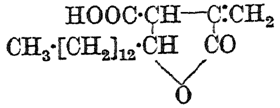
Suggested structure

$C_{25}H_{32}O_8$ MW, 460

Isolated from the dried female flowers of *Hagenia abyssinica* (*Brayera anthelmintica*). Needles from $CHCl_3$–EtOH. M.p. 182°. Sol. Et_2O, $CHCl_3$, Me_2CO, AcOEt. Spar. sol. EtOH, pet. ether. Insol. H_2O. $[\alpha]_D$ + 8·0° in $CHCl_3$. Possesses no vermicidal properties. Sol. conc. H_2SO_4 with pale green col. ⟶ deep red on warming. Alc. $FeCl_3$ ⟶ reddish brown col.

Hems, Todd, *J. Chem. Soc.*, 1937, 562.
Birch, Todd, *J. Chem. Soc.*, 1952, 3102.

Protolichesteric Acid



$C_{19}H_{32}O_4$ MW, 324

l-.

Obtained from Japanese sub-alpine moss. Plates from AcOH. M.p. 107·5°. $[\alpha]_D^{27}$ − 12·71°.

Semicarbazone: m.p. 140°.

Pyrazoline deriv.: plates from pet. ether. M.p. 54–5°. $[\alpha]_D^{18}$ − 183·1°.

d-.

Obtained from European Iceland moss. Plates from AcOH. M.p. 106°. $[\alpha]_D^{20}$ + 12·07°.

Pyrazoline deriv.: plates. M.p. 54–5°. $[\alpha]_D^{18}$ + 190·6°.

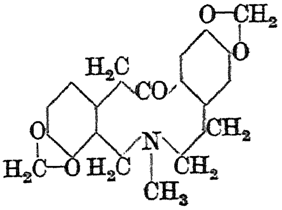
l- (*Allo*-).

Plates from AcOH. M.p. 88°. $[\alpha]_D^{20}$ − 49·53°.

Pyrazoline deriv.: plates from pet. ether. M.p. 68–9°. $[\alpha]_D^{18}$ − 73·69°.

Asahina, Asano, *J. Pharm. Soc. Japan*, 1927, **539**, 1.
Asano, Kanematsu, *Ber.*, 1932, **65**, 1175.
Asahina, Yanagita, *Ber.*, 1936, **69**, 120.
Asahina, Yasue, *Ber.*, 1937, **70**, 1053.

Protopine (*Fumarine, corydalis-C*)



$C_{20}H_{19}O_5N$ MW, 353

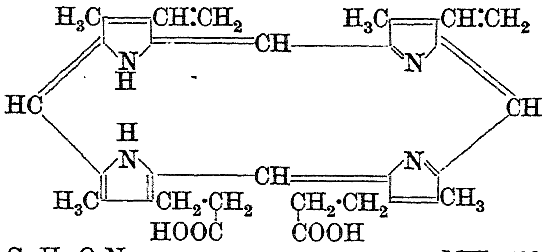
Widely distributed in the *Papaveraceæ* and *Fumariaceæ*. Prisms from MeOH. M.p. 208°. Sol. $CHCl_3$. Spar. sol. MeOH, EtOH, Me_2CO, C_6H_6. Insol. H_2O. AcOH sol. ⟶ deep bluish-violet col. with H_2SO_4.

Methiodide: cryst. from MeOH. M.p. 217°.

Methosulphate: prisms from MeOH. M.p. 252°.

Perkin, *J. Chem. Soc.*, 1916, **109**, 1023.
Haworth, Perkin, *J. Chem. Soc.*, 1926, 1769.

Protoporphyrin



$C_{34}H_{34}O_4N_4$ MW, 562

Deep red cryst. Does not melt below 300°. Sol. EtOH, Py, AcOEt, AcOH. Spar. sol. Et_2O, pet. ether. Insol. H_2O. Obtained from hæmatin by red. with $SnCl_2$ in MeOH.

Mono-Me ester: m.p. 224–6°.

Di-Me ester: m.p. 233°.

Acetyl: dark violet cryst. M.p. 231°.

Monopicrate: dark violet cryst. M.p. 180°.

Dipicrate: violet cryst. M.p. 157°.

Styphnate: pale violet-grey cryst. M.p. 234°.

Diflavianate: violet cryst. M.p. 211°.

Triflavianate: violet-brown cryst. with yellow surface lustre. M.p. 215°.

Fischer, Lindner, *Z. physiol. Chem.*, 1925, **142**, 141.
Fischer, Pützer, *Z. physiol. Chem.*, 1926, **154**, 39.
Hamsik, *Z. physiol. Chem.*, 1929, **180**, 308; 1931, **196**, 195.
Fischer, Bäumler, *Ann.*, 1929, **468**, 58.
Fischer, Hummel, Treibs, *Ann.*, 1929, **471**, 146.
Treibs, *Ann.*, 1929, **476**, 1.
Fischer, Eckoldt, *Ann.*, 1940, **544**, 138.
Grinstein, *J. Biol. Chem.*, 1947, **167**, 515.

Protostephanine

$C_{21}H_{25}O_4N$ MW, 355

Alkaloid from *Stephnia japonica*, Miers. Prisms + $1\frac{1}{2}$MeOH from MeOH. M.p. 75°, 90–5° solvent free. Optically inactive. Contains 1 $N{\cdot}CH_3$ and 4 OCH_3 groups.

B,HCl: decomp. at 150°.

B_2,H_2PtCl_6: orange prisms. Decomp. at 223°.

Methiodide: prisms from H_2O. M.p. 220–1°.

Methomethylsulphate: prisms from H_2O. Sinters at 227°. M.p. 235°.

Kondo, Watanabe, *J. Pharm. Soc. Japan*, 1938, **58**, 46.

Protoveratridine

$C_{32}H_{51}O_9N$ MW, 593

Alkaloid from *Veratrum album*. Cryst. M.p. 266–7° corr. Spar. sol. all org. solvents.

B,HCl: cryst. from EtOH. M.p. 243–5° corr. decomp.

Methylethylglycollic ester: *see* Germerine.

Picrate: yellow needles from Me_2CO–Et_2O. Decomp. at 244–6° corr.

Chloroplatinate: cryst. from EtOH.Aq. Decomp. at 195–200° corr.

Poethke, *Arch. Pharm.*, 1937, **275**, 571.
Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., p. 601, (Reinhold Publishing Corporation, New York).

Protoveratrine

$C_{39}H_{61}O_{13}N$ MW, 751

Alkaloid from *Veratrum album*. Cryst. from EtOH. M.p. 255–6° corr. decomp. $[\alpha]_D^{20} - 9{\cdot}1°$ in $CHCl_3$.

B,HCl: plates. M.p. 234–6° corr. decomp.

B,HBr: prisms. M.p. 230–2° corr. decomp.

B,HI: prisms. M.p. 247–8° corr. decomp.

B,HSCN: prisms. M.p. 221–3° corr. decomp.

$B,HAuCl_4$: yellow cryst. Decomp. at 199° corr.

Picrate: yellow needles. M.p. 216–20° corr. decomp.

Poethke, *Arch. Pharm.*, 1937, **275**, 571.
Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., p. 601, (Reinhold Publishing Corporation, New York).

Prulaurasine (dl-*Mandelonitrile*-d-β-*glucoside, laurocerolin*)

$$C_6H_5{\cdot}CH<\begin{matrix}O{\cdot}C_6H_{11}O_5\\CN\end{matrix}$$

$C_{14}H_{17}O_6N$ MW, 295

Occurs in leaves of common cherry laurel (*Prunus laurocerasus*). Prisms or needles. M.p. 122–122·5°. $[\alpha]_D - 54°$. Sol. H_2O, MeOH. Insol. Et_2O. Hyd. ⟶ *dl*-mandelonitrile + glucose.

Tetra-acetyl: needles. M.p. 120–3°. Sol. EtOH, Me_2CO, $CHCl_3$, AcOEt, C_6H_6. Insol. ligroin.

Hérissey, *Compt. rend.*, 1905, **41**, 959.
Caldwell, Courtauld, *J. Chem. Soc.*, 1907, **91**, 671.
Fischer, Bergmann, *Ber.*, 1917, **50**, 1062.

Prulaurasinic Acid (dl-*Mandelic acid* β-d-*glucoside*)

$$C_6H_5{\cdot}CH<\begin{matrix}O{\cdot}C_6H_{11}O_5\\COOH\end{matrix}$$

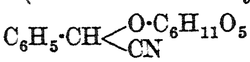
$C_{14}H_{18}O_8$ MW, 314

White hygroscopic powder. Cryst. with 1EtOH. $[\alpha]_D^{11} - 28{\cdot}17°$ to $- 33{\cdot}18°$. Hyd. by emulsin. Does not reduce Fehling's.

Tetra-acetyl: needles. M.p. 130–50°. $[\alpha]_D^{15} - 36{\cdot}97°$ to $- 43{\cdot}46°$. *Et ester*: needles from EtOH. M.p. 102–9°. $[\alpha]_D - 33°$ to $- 40{\cdot}1°$ in C_6H_6. Sol. EtOH, Me_2CO, AcOEt, C_6H_6. Spar. sol. Et_2O, pet. ether.

Karrer, Nägeli, Weidmann, *Helv. Chim. Acta*, 1919, **2**, 257.
Fischer, Bergmann, *Ber.*, 1917, **50**, 1053.

Prunasine (d-*Mandelonitrile*-d-*glucoside*)



$C_{14}H_{17}O_6N$ MW, 295

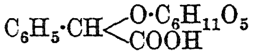
Occurs in *Prunus laurocerasus*, *P. padus* and *P. cerasus*. Needles from AcOEt. M.p. 147–50°. $[\alpha]_D$ − 27°. Sol. H_2O, MeOH, Me_2CO. Alkalis ⟶ prulaurasine. HCl ⟶ *d*-mandelic acid.

Tetra-acetyl: needles from EtOH. M.p. 125–6°. $[\alpha]_D^{22}$ − 52·5°.

Fischer, *Ber.*, 1895, **28**, 1508.
Caldwell, Courtauld, *J. Chem. Soc.*, 1907, **91**, 666, 671.
Auld, *J. Chem. Soc.*, 1908, **93**, 1276.
Fischer, Bergmann, *Ber.*, 1917, **50**, 1047.

Note.—All references state prunasine is derived from *l*-mandelonitrile, but see note under Mandelic Acid concerning the confusion which existed between the *d*- and *l*-forms prior to 1923.

Prunasinic Acid (d-*Mandelic acid*-β-d-*glucoside*)



$C_{14}H_{18}O_8$ MW, 314

$[\alpha]_D^{15}$ − 138·6°. Sol. H_2O.

Tetra-acetyl: needles. M.p. 132°. $[\alpha]_D^{15}$ − 82·40° in EtOH.

Karrer, Nägeli, Weidmann, *Helv. Chim. Acta*, 1919, **2**, 257.
See note under Prunasine.

Prunetin.
See under Genistein.

Prunetol.
See Genistein.

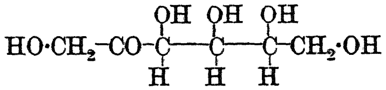
Prunol.
See Ursolic Acid.

Prussic Acid.
See Hydrocyanic Acid.

Pseudaconine.
See ψ-Aconine.

Pseudaconitine.
See ψ-Aconitine.

Psicose (2-*Ketoribohexose*, ψ-*fructose*, *allulose*)



$C_6H_{12}O_6$ MW, 180

d-.

Syrup. Not fermentable. $[\alpha]_D^{20}$ + 3·1° in H_2O.

Diacetone deriv.: m.p. 57–58·5°. B.p. 104–5°/0·3 mm. $[\alpha]_D^{20}$ − 98·2° in Me_2CO.

Phenylosazone: m.p. 173–4° decomp. (identical with *d*-allosazone). $[\alpha]_D^{20}$ − 19·2° in EtOH.

l-.

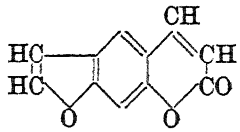
Syrup. $[\alpha]_D^{20}$ − 3·3° in H_2O.

Diacetone deriv.: m.p. 56·5–57°. $[\alpha]_D^{20}$ + 99° in Me_2CO.

Phenylosazone: m.p. 173–4° decomp. (identical with *l*-allosazone). $[\alpha]_D^{20}$ + 19° in EtOH.

Steiger, Reichstein, *Helv. Chim. Acta*, 1935, **18**, 790; 1936, **19**, 184.
Wolfrom, Thompson, Evans, *J. Am. Chem. Soc.*, 1945, **67**, 1793.

Psoralene (*Furo-coumarin*)



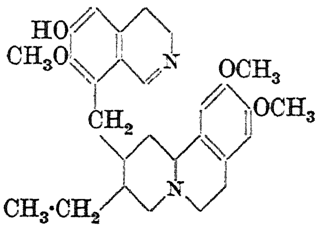
$C_{11}H_6O_3$ MW, 186

Obtained from *Psoralea corylifolia*, Linn, Needles from H_2O. M.p. 171°. Sol. EtOH. $CHCl_3$. Spar. sol. H_2O, Et_2O, pet. ether.

Späth, Manjunath, Pailer, Jois, *Ber.*, 1936, **69**, 1087.
Jois, Manjunath, Rao, *J. Indian Chem. Soc.*, 1933, **10**, 41.
Horning, Reisner, *J. Am. Chem. Soc.*, 1950, **72**, 1514.

Psychosine.
See under Sphingosine.

Psychotrine



$C_{28}H_{36}O_4N_2$ MW, 464

Obtained from ipecacuanha. Colourless odourless prisms + $4H_2O$ from EtOH.Aq. or Me_2CO.Aq. M.p. anhyd. 122°. Sol. Et_2O. Very sol. EtOH, Me_2CO, $CHCl_3$. Spar. sol. cold H_2O. Insol. cold C_6H_6, ligroin. $[\alpha]_D^{15}$ + 69·3° in EtOH. Alc. sol. turns litmus blue. Bitter taste. Blue fluor. in some sols. $FeCl_3$ ⟶ reddish-brown to bluish-black col.

Me ether: prisms from Et_2O. M.p. 123–4°. $[\alpha]_D$ + 43·2° in EtOH. *Picrate*: plates from Me_2CO. Softens at 142°. Slowly melts up to 175°. *B,H₂SO₄*: prisms + $7H_2O$ from H_2O. M.p. 247°. $[\alpha]_D$ + 44·4° in H_2O. *B,HBr*: yellow needles from H_2O. M.p. anhyd. 190–200°. $[\alpha]_D$ (anhyd.) + 48·0° in H_2O.

Hydrogen oxalate: needles from EtOH. M.p. 130–45°.

Dibenzoate: amorph. powder. Sinters at 120°. M.p. 132–5°. Very sol. Et_2O, EtOH.

Brindley, Pyman, *J. Chem. Soc.*, 1927, 1067.
Pyman, *J. Chem. Soc.*, 1917, **111**, 431.
Hesse, *Ann.*, 1914, **405**, 34.
Openshaw, Wood, *J. Chem. Soc.*, 1952, 391.

Psyllaic Acid.
See Psyllostearic Acid.

Psylla-alcohol.
See Psyllostearyl Alcohol.

Psyllostearic Acid (*Psyllaic acid*)

$C_{33}H_{66}O_2$ MW, 494

Cryst. M.p. 94–5°. Sol. hot EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. cold H_2O, EtOH, Et_2O.

Sundwik, *Z. physiol. Chem.*, 1911, **72**, 455; 1907, **53**, 365.

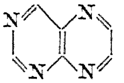
Psyllostearyl Alcohol (*Psylla-alcohol*)

$C_{33}H_{68}O$ MW, 480

Obtained with psyllostearic acid by hyd. of wax from *Psylla alni*. Cryst. from Me_2CO. M.p. 69–69·5°. Very sol. hot Me_2CO. Soda-lime at 360–70° ⟶ psyllostearic acid.

Sundwik, *Z. physiol. Chem.*, 1911, **72**, 455; 1907, **53**, 365.

Pteridine (*Pyrimido*-[4, 5-b]-*pyrazine*)



$C_6H_4N_4$ MW, 132

Parent compound of a group of natural products, including the butterfly wing pigments and folic acid. Yellow plates from C_6H_6. M.p. 138–138·5°. Sublimes at 125–30°/20 mm., giving denser yellow cryst., m.p. 139·5–140°.

N-*Oxide*: white cryst. from MeOH. Decomp. at about 350°.

Schöpf, Reichert, *Ann.*, 1941, **548**, 82.
Simpson, *Annual Reports on the Progress of Chemistry* (London), 1946, XLIII, 250.
Elvidge, *Annual Reports on the Progress of Chemistry* (London), 1948, XLV, 226.
Albert, Brown, Cheeseman, *J. Chem. Soc.*, 1951, 474.
Albert, *Quarterly Reviews*, 1952, **6**, 197.

Pterobilin

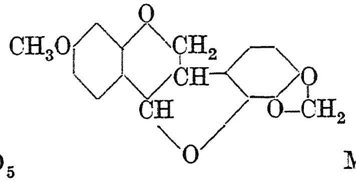
$C_{33}H_{34}O_6N_4$ MW, 582

Wing pigment of butterflies *Pteris brassicae, P. rapae, P. napi, Catopsilia rurina* and *C. statira*. Dark blue rods from AcOEt. Does not melt or decomp. at 310°. Conc. HNO_3 on $CHCl_3$ sol. ⟶ blue col. eventually becoming colourless after passing through green, orange, yellow, etc.

Di-Me ester: $C_{35}H_{38}O_6N_4$. MW, 610. M.p. 234°. *Zn salt*: green needles. Decomp. above 300°.

Wieland, Tartter, *Ann.*, 1940, **545**, 197.

Pterocarpine



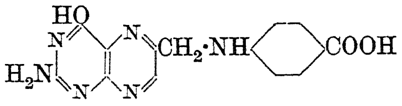
$C_{17}H_{14}O_5$ MW, 298

Obtained from red sandalwood. Needles from CCl_4. M.p. 165°. Insol. H_2O, cold EtOH, CS_2, acids, alkalis. Spar. sol. Et_2O. $[\alpha]_D^{20}$ – 220·1°.

Dinitrophenylhydrazone: dark brown needles. M.p. 305° decomp.

Raudnitz, Perlmann, *Ber.*, 1935, **68**, 1862.
Leonhardt, Fay, *Arch. Pharm.*, 1935, **273**, 53.
Dieterle, Leonhardt, *Arch. Pharm.*, 1929, **267**, 81.
McGookin, Robertson, Whalley, *J. Chem. Soc.*, 1940, 787.

Pteroic Acid

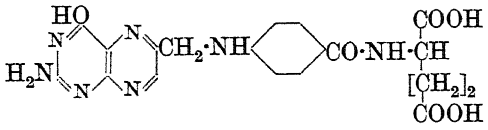


$C_{14}H_{12}O_3N_6$ MW, 312

Needles from H_2O. Mod. sol. hot H_2O, MeOH, EtOH, AcOH, Py. Insol. Me_2CO, $CHCl_3$, cold H_2O.

Hutchings *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 1.

Pteroylglutamic Acid (*Folic acid, vitamin* B_c, *vitamin M*, L. casei *factor*)

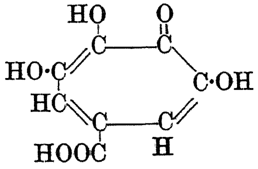


$C_{19}H_{19}O_6N_7$ MW, 441

Pale lemon yellow to light orange needles from H_2O at pH 3. Chars above 250°. Mod. sol. hot H_2O, EtOH, AcOH, PhOH, Py. Spar. sol. cold H_2O. Insol. Me_2CO, $CHCl_3$.

Pfiffner *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 1476.
Hutchings *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 1–28.
Simpson, *Annual Reports on the Progress of Chemistry* (London), 1946, XLIII, 257.

Puberulic Acid



$C_8H_6O_6$ MW, 198

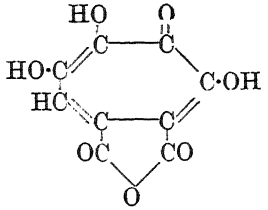
Obtained from cultures of the moulds *Penicillium puberulum* and *Penicillium aurantio-virens*. Possesses antibiotic properties. Colourless plates from MeOH. M.p. 318–20°. Sol. H_2O, MeOH.

Diacetyl: m.p. 212°.

Tetra-Me ether: m.p. 112°.

Birkinshaw, Raistrick, *Biochem. J.*, 1932, **26**, 441.
Barger, Dorrer, *Biochem. J.*, 1934, **28**, 11.
Corbett, Hassall, Johnson, Todd, *J. Chem. Soc.*, 1950, 1.
Corbett, Johnson, Todd, *ibid.*, 6.

Puberulonic Acid



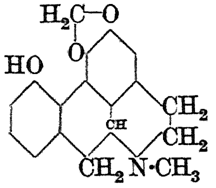
$C_9H_4O_7$ MW, 224

Obtained from cultures of the moulds *Penicillium puberulum* and *Penicillium aurantio-virens*. Possesses antibiotic properties. Fine yellow needles from diethyl malonate. M.p. 298° decomp.

Py add. comp.: yellow needles from AcOH. M.p. 257–8° decomp.

Birkinshaw, Raistrick, *Biochem. J.*, 1932, **26**, 441.
Barger, Dorrer, *Biochem. J.*, 1934, **28**, 11.
Johnson, Sheppard, Todd, *J. Chem. Soc.*, 1951, 1139.

Pukateine



$C_{18}H_{17}O_3N$ MW, 295

l-.

Obtained from the bark of the pukatea (*Laurelia Novae Zealandiae*). Cryst. from Et_2O. M.p. 200°. B.p. 210–15°/2 mm. Sol. EtOH, Et_2O, $CHCl_3$. Very sol. pyridine. Spar. sol. pet. ether. Insol. H_2O. $[\alpha]_D^{15}$ − 220° in EtOH. H_2SO_4 ⟶ orange sol. changing to red and violet on warming. HNO_3 ⟶ vermilion sol., orange on warming. $K_2Cr_2O_7$ in H_2SO_4 ⟶ purple col., excess of reagent ⟶ green col. Has an action quantitatively similar to morphine on central nervous system.

Acetyl: *methiodide*, needles from Me_2CO. M.p. 245°.

Me ether: cryst. from dil. EtOH. M.p. 137°. $[\alpha]_D$ − 261°. H_2SO_4 ⟶ orange to mauve col. HNO_3 ⟶ brown sol. *B,HCl*: cryst. from EtOH. M.p. 281°. *B,HBr*: cryst. from EtOH. M.p. 234°. d-*Tartrate*: needles from EtOH.Aq. M.p. 234°. $[\alpha]_D^{20}$ − 149·1° in EtOH. *Methiodide*: cryst. M.p. 240–1°.

d-.

Me ether: cryst. from EtOH.Aq. M.p. 136°. $[\alpha]_D^{20}$ + 256·4° in EtOH. l-*Tartrate*: cryst. from EtOH. M.p. 225°. $[\alpha]_D^{20}$ + 147·5°.

Barger, Schlittler, *Helv. Chim. Acta*, 1932, **15**, 381.
Barger, Girardet, *Helv. Chim. Acta*, 1931, **14**, 481.
Aston, *J. Chem. Soc.*, 1910, **97**, 1381.

Pulcheremodin

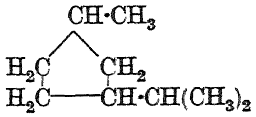
$C_{15}H_{10}O_5$ MW, 270

From root of *Rumex pulcher*, Linn. Orange needles. M.p. 251°. Readily sol. EtOH, Et_2O, Py. Sol. $CHCl_3$, Me_2CO, AcOH. Mod. sol. MeOH, CS_2. Spar. sol. CCl_4, C_6H_6. H_2SO_4 ⟶ red col.

Triacetate: yellow needles. M.p. 194°. Sol. EtOH, AcOH, C_6H_6. H_2SO_4 ⟶ carmine red col.

Emmanuel, *Chem. Zentr.*, 1918, I, 564.

Pulegan (*3-Methyl-1-isopropylcyclopentane*)

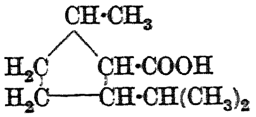


C_9H_{18} MW, 126

Oil with terpene odour. B.p. 142–4°. D^{22} 0·7730. n_D 1·4236.

Wallach, *Ann.*, 1912, **392**, 58.

Puleganic Acid (*Dihydropulegenic acid*, *5-methyl-2-isopropylcyclopentane-carboxylic acid*)



$C_{10}H_{18}O_2$ MW, 170

Colourless viscous oil. F.p. − 18 to − 19°. B.p. 152°/25 mm., 139°/12 mm. D_4^{20} 0·9642. n_D^{24} 1·4524. $[\alpha]_D^{20}$ − 0·36°.

Me ester: $C_{11}H_{20}O_2$. MW, 184. B.p. 91°/13 mm.

Et ester: $C_{12}H_{22}O_2$. MW, 198. B.p. 145°/4 mm. $D_4^{11\cdot8}$ 0·9178. $n_\alpha^{11\cdot8}$ 1·4405.

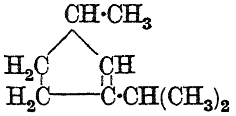
Chloride: $C_{10}H_{17}OCl$. MW, 188·5. B.p. 89–90°/11 mm.

Amide: $C_{10}H_{19}ON$. MW, 169. Cryst. from

EtOH.Aq. or AcOEt–ligroin. M.p. 150–1°. $[\alpha]_D^{20}$ + 4·8° in MeOH.
Nitrile: $C_{10}H_{17}N$. MW, 151. B.p. 103°/13 mm. $D_4^{16\cdot2}$ 0·8814. $n_\alpha^{16\cdot2}$ 1·4475.
Anilide: cryst. from EtOH.Aq. M.p. 149–50°.

Rupe, Schäfer, *Helv. Chim. Acta*, 1928, **11**, 467.
Wallach, *Ann.*, 1918, **414**, 237.

Pulegene (3-*Methyl*-1-*isopropylcyclopentene*)

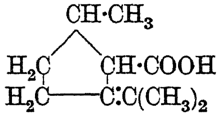


C_9H_{16} MW, 124

Oil. B.p. 138–9°, 39–41°/16 mm. D^{22} 0·791. n_D^{22} 1·4380.
Nitrosochloride: cryst. M.p. 74–5°.

Wallach, Collmann, Thede, *Ann.*, 1903, **327**, 131.

Pulegenic Acid (5-*Methyl*-2-*isopropylidene-cyclopentane-carboxylic acid*)



$C_{10}H_{16}O_2$ MW, 168

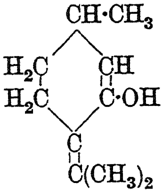
Pale yellow oil. B.p. 144–6°/12 mm. D_4^{20} 1·0050. n_D^{20} 1·4754. $[\alpha]_D^{20}$ + 48·18°. MeOH–HCl ⟶ hydrochloride of Me ester. Aniline at 200° ⟶ pulegene.
Me ester: $C_{11}H_{18}O_2$. MW, 182. B.p. 98–101°/10 mm. D^{20} 0·97. n_D^{20} 1·4665. *Hydrochloride*: m.p. 15–16°. B.p. 114°/12 mm.
Amide: $C_{10}H_{17}ON$. MW, 167. Needles from H_2O or EtOH.Aq. M.p. 123°. Very sol. EtOH, Et_2O. $[\alpha]_D^{18}$ + 29·05° in MeOH. Red. ⟶ amide of puleganic acid.
Nitrile: $C_{10}H_{15}N$. MW, 149. B.p. 218–20°. n_D^{22} 1·47047.
Anilide: needles from Et_2O–pet. ether. M.p. 124°. B.p. 200°/10 mm. Very sol. EtOH, Et_2O. Spar. sol. pet. ether.
p-*Toluidide*: needles from Et_2O–pet. ether. M.p. 143°.

Rüpe, Schäfer, *Helv. Chim. Acta*, 1928, **11**, 466.
Wallach, *Ann.*, 1918, **414**, 242.

β-Pulegenic Acid (*Isopulegenic acid*).
Differs from pulegenic acid in position of double bond. Oil. B.p. 142–5°/11 mm. D^{20} 0·9975. n_D 1·4747. $[\alpha]_D^{22}$ + 32·7° in Et_2O.
Amide: $C_{10}H_{17}ON$. MW, 167. Needles from MeOH.Aq. M.p. 152°. Red. ⟶ amide of puleganic acid.

Wallach, *Ann.*, 1918, **414**, 242.

Pulegenol (*Enol form of pulegone*, $\Delta^{2,4(8)}$-p-*menthadienol*-3)

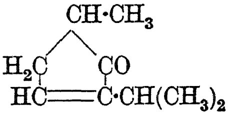


$C_{10}H_{16}O$ MW, 152

Oil. B.p. 85°/6 mm. D_4^{15} 0·916. n_D^{20} 1·48312. $[\alpha]_D$ + 24·6°. Dist. in steam or action of alkalis ⟶ pulegone.
Acetyl: oil.
Benzoyl: cryst. M.p. 230°.
Et ether: $C_{12}H_{20}O$. MW, 180. B.p. 97–97·5°/12 mm. D_0^{20} 0·9047.

Grignard, Blanchon, *Bull. soc. chim.*, 1931, **49**, 23.
Grignard, Savard, *Bull. soc. chim. Belg.*, 1927, **36**, 97; *Compt. rend.*, 1924, **179**, 1573.

Pulegenone (4-*Methyl*-1-*isopropylcyclopentenone*-5)

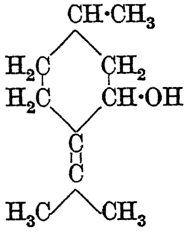


$C_9H_{14}O$ MW, 138

Oil. B.p. 188·5–189°. D_0^{20} 0·9144. n_D^{20} 1·4660.
Oxime: b.p. 237–42°, 123–6°/15 mm. *Benzoyl*: cryst. from MeOH. M.p. 104–5°.
Semicarbazone: cryst. from MeOH. M.p. 183–4°.

Wallach, Grote, *Ann.*, 1919, **418**, 50.
Calas, *Bull. soc. chim.*, 1939, **6**, 1374.

Pulegol ($\Delta^{4(8)}$-p-*Menthenol*-3, 1-*methy*-4-*isol-propylidenecyclohexanol*-3)



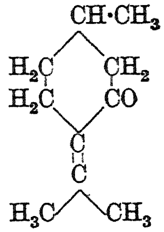
$C_{10}H_{18}O$ MW, 154

Two forms are described. (i) Obtained by Na–EtOH reduction of pulegone. Needles. M.p. 46–7°. B.p. 209–10°. $[\alpha]_D$ − 54·1° in EtOH. *Hydrogen phthalate*: needles from pet. ether. M.p. 212°. $[\alpha]_D$ − 86·8° in EtOH. (ii) Obtained by reduction of pulegone with aluminium isopropylate. B.p. 91·5°/12 mm. D_4^{18} 0·909. n_D^{18} 1·4714. $[\alpha]_{5461}^{18}$ + 80·09°.

Doeuvre, Perret, *Bull. soc. chim.*, 1935, **2**, 298.
Paolini, *Atti accad. Lincei*, 1919, **28**, 190, 236.

β-Pulegomenthol.
See Neomenthol

Pulegone ($\Delta^{4(8)}$-p-*Menthenone*-3)



$C_{10}H_{16}O$ MW, 152

In oils from *Mentha pulegium*, *Hedeoma pulegoides*, *Mentha sylvestris*, etc. Colourless oil with pleasant peppermint odour. B.p. 224°, 151–3°/100 mm., 103°/17 mm., 56–7°/0·9 mm. D_4^{19} 0·937. n_D^{19} 1·4880. $[\alpha]_D^{20}$ + 23·4°. Heat of comb. C_v 1411·6 Cal. Readily purified through $NaHSO_3$ comp. Ox. ⟶ acetone + *d*-2-methyl-adipic acid.

Hydrochloride: cryst. from ligroin. M.p. 24–5°.

Hydrobromide: cryst. from EtOH.Aq. M.p. 40·5°. Very sol. EtOH, Et_2O. $[\alpha]_D$ − 33·88° in EtOH.

Nitrosite: needles from EtOH. M.p. 68–9°. Sol. EtOH, AcOH, $CHCl_3$.

Dinitroso deriv.: m.p. 81·5°.

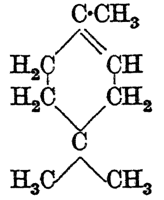
2 : 4-*Dinitrophenylhydrazone*: red plates from pet. ether. M.p. 142°.

Semicarbazone: prisms from EtOH. M.p. 174°. Mod. sol. EtOH.

Phenylsemicarbazone: needles from EtOH. M.p. 132–3°.

Doeuvre, Perret, *Bull. soc. chim.*, 1935, 2, 298.
Kon, *J. Chem. Soc.*, 1930, 1616.
Baeyer, Henrich, *Ber.*, 1895, **28**, 652.
Naves, *Helv. Chim. Acta*, 1948, **31**, 893.

Pulenene (1 : 4 : 4-*Trimethylcyclohexene*)



C_9H_{16} MW, 124

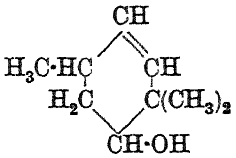
Liq. with pleasant odour. B.p. 139·5–140·5°, 36·3–37·3°/14 mm. $D_4^{18\cdot8}$ 0·8032. $n_D^{23\cdot2}$ 1·444. Ox. ⟶ 2 : 2-dimethyl-4-acetyl-*n*-valeric acid. H_2SO_4 ⟶ reddish-blue col.

Nitrosochloride: rhombohedra from EtOH, needles from AcOEt. M.p. 118–22°.

Other pulenenes are described in the literature. Little data is given and they are of doubtful purity.

Auwers, Lange, *Ann.*, 1915, **409**, 167.
Wallach, Kempe, *Ann.*, 1903, **329**, 89.

βγ-Pulenenol (3 : 3 : 6-*Trimethylcyclohexenol*-4)

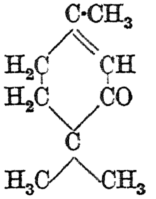


$C_9H_{16}O$ MW, 140

Oil with peppermint odour. B.p. 189°/754 mm., 82–5°/15 mm. $D_4^{18\cdot5}$ 0·9209. $n_D^{18\cdot5}$ 1·47398.

Auwers, Hessenland, *Ber.*, 1908, **41**, 1807.

αβ-Pulenenone (1 : 4 : 4-*Trimethylcyclohexenone*-3)



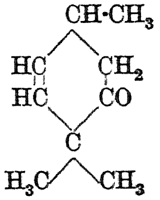
$C_9H_{14}O$ MW, 138

Oil with pleasant odour. B.p. 208°/753 mm., 86–8°/15 mm., 81°/13 mm. D_4^{25} 0·927. $D_4^{16\cdot5}$ 0·9317. n_D^{20} 1·4798, $n_D^{16\cdot5}$ 1·47958. Na–EtOH ⟶ pulenol.

Semicarbazone: cryst. from MeOH or EtOH. M.p. 201°. Very sol. hot MeOH. Mod sol. cold MeOH.

Auwers, Hessenland, *Ber.*, 1908, **41**, 1812.
Buchman, Sargent, *J. Org. Chem.*, 1942, 7, 144.

βγ-Pulenenone (3 : 3 : 6-*Trimethylcyclohexenone*-4)



$C_9H_{14}O$ MW, 138

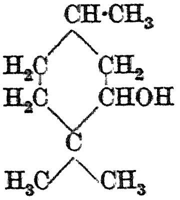
Oil with peppermint odour. B.p. 172–4°, 63–5°/16 mm. $D_4^{17\cdot7}$ 0·9055. $n_D^{17\cdot7}$ 1·45582.

Dichloride: oil. B.p. 121–122·5°/13 mm. D_4^{22} 1·2008. n_D^{22} 1·50002.

Semicarbazone: needles from MeOH.Aq. or C_6H_6–ligroin. M.p. 127°.

Auwers, Hessenland, *Ber.*, 1908, **41**, 1812.

Pulenol (2 : 2 : 5-*Trimethylcyclohexanol*)

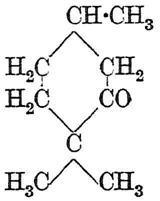


$C_9H_{18}O$ MW, 142

Oil. B.p. 187–9°, 90–2°/23 mm. D^{20} 0·8955. n_D^{20} 1·4569. CrO_3 ⟶ pulenone.
Phenylurethane: cryst. from EtOH.Aq. M.p. 92° (82–5°).

Auwers, Hessenland, *Ber.*, 1908, **41**, 1814.
Wallach, Kempe, *Ann.*, 1903, **329**, 87.

Pulenone (2 : 2 : 5-*Trimethylcyclohexanone*)



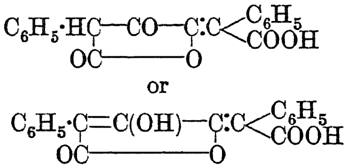
$C_9H_{16}O$ MW, 140

d-.
B.p. 183°. Spar. sol. H_2O. D^{21} 0·8925. n_D^{21} 1·44506. Strongly dextrorotatory.
Oxime: needles. M.p. 94–5°. B.p. 117°/12 mm.
Semicarbazone: m.p. 169–70°.

dl-.
B.p. 182–4°, 90–2°/39 mm. D^{24} 0·8871. n_D^{24} 1·4432.
Oxime: cryst. from EtOH. M.p. 93·5°.
Semicarbazone: m.p. 176–7° (169–70°).

Auwers, Hessenland, *Ber.*, 1908, **41**, 1814.
Wallach, Kempe, *Ann.*, 1903, **329**, 86.
Cornubert, Humeau, *Bull. soc. chim.*, 1931, **49**, 1469.

Pulvinic Acid (*Pulvic acid*)

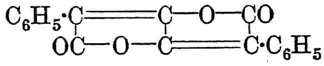


$C_{18}H_{12}O_5$ MW, 308

Orange powder from Et_2O or $CHCl_3$, prisms from C_6H_6, yellow cryst. + 1MeOH from MeOH, yellowish-red cryst. + 1EtOH from EtOH. M.p. 216–17°. Very sol. EtOH, hot AcOH. Sol. H_2O and pptd. by acids. Spar. sol. Et_2O, $CHCl_3$, C_6H_6. Heat. above m.p., or with Ac_2O or CH_3COCl ⟶ lactone. Ox. ⟶ oxalic and benzoylformic acids.
Me ester: *see* Vulpinic Acid.
Et ester: $C_{20}H_{16}O_5$. MW, 336. Yellow tablets from EtOH. M.p. 127–8°. Heat above m.p. ⟶ lactone. *Acetyl*: needles. M.p. 143–4°. *Me ether*: needles from EtOH. Prisms from AcOH. M.p. 150–1°.
Propyl ester: $C_{21}H_{18}O_5$. MW, 350. Yellow needles or plates from $CHCl_3$. M.p. 134°. Very sol. $CHCl_3$, C_6H_6. Spar. sol. EtOH. *Me ether*: needles. M.p. 121–2°.
Amide: $C_{18}H_{13}O_4N$. MW, 307. Yellow prisms from C_6H_6, yellow plates from AcOH. Sinters at 220–1°, m.p. 226°. Very sol. Me_2CO. Mod. sol. Et_2O, C_6H_6. Spar. sol. EtOH. Insol. H_2O. *NH_4 salt*: needles. M.p. 218°. Very sol. EtOH. Spar. sol. cold H_2O. *Me ether*: cryst. from MeOH. M.p. 216–17°.
Methylamide: plates from EtOH–C_6H_6. M.p. 237°.
Dimethylamide: prisms. M.p. 211°.
Nitrile: $C_{18}H_{11}O_3N$. MW, 289. Reddish-yellow needles from EtOH. Sinters at 190°, m.p. 193–4°. *Acetyl*: yellow needles from EtOH. M.p. 141–2°. Very sol. Et_2O. *Benzoyl*: pale yellow needles from EtOH. M.p. 168–168·5°. Insol. H_2O.
Anilide: cryst. from AcOH. M.p. 187–8°.
1-*Naphthylamide*: reddish-yellow plates from toluene. M.p. 211–12°.
2-*Naphthylamide*: reddish-yellow cryst. from toluene. M.p. 192°.

Asano, Yameda, *Ber.*, 1935, **68**, 1569.
Koller, Pfeiffer, *Monatsh.*, 1933, **62**, 164.
Karrer, Gehrekens, Heuss, *Helv. Chim. Acta*, 1926, **9**, 456.
Mazza, *Chem. Zentr.*, 1926, II, 1037.
Volhard, *Ann.*, 1894, **282**, 14.
Schenck, *ibid.*, 39.

Pulvinic Acid Lactone (*Pulvic acid lactone*)



$C_{18}H_{10}O_4$ MW, 290

Found in *Sticta aurata* Ach, *Candelaria medians*, etc. Pale yellow needles from C_6H_6 or AcOH. M.p. 222–4°. Sol. hot $CHCl_3$, C_6H_6, AcOH, Me_2CO. Spar. sol. hot EtOH. Insol. H_2O. Insol. cold aq. alkalis. Sublimes in needles. Hyd. ⟶ pulvinic acid. NH_4OH ⟶ NH_4 salt of pulvinic acid amide. KOH–MeOH ⟶ vulpinic acid. Aniline ⟶ pulvinic acid anilide.
A second lactone of pulvinic acid is described in the literature. Cryst. from AcOH. M.p. 124–5°.

Asano, Yameda, *Ber.*, 1935, **68**, 1569.
Koller, Pfeiffer, *Monatsh.*, 1933, **62**, 164.
Karrer, Gehrekens, Heuss, *Helv. Chim. Acta*, 1926, **9**, 456.
Mazza, *Chem. Zentr.*, 1926, II, 1037.
Volhard, *Ann.*, 1894, **282**, 14.
Schenck, *ibid.*, 39.

Punicic Acid

$CH_3\cdot[CH_2]_3\cdot[CH{:}CH]_3\cdot[CH_2]_7\cdot COOH$

$C_{18}H_{30}O_2$ MW, 278

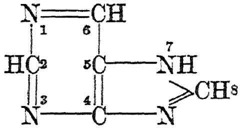
Geometrical isomer of elæostearic acid isolated from oil of pomegranate seeds. Cryst. from pet. ether. M.p. 44°. D_4^{50} 0·9027. n_D^{50} 1·5114. Readily absorbs atmospheric oxygen. Irradiation of xylene sol. for 4 hours ⟶ β-elæostearic

acid. H(+ Pt) ⟶ stearic acid. $KMnO_4$ ⟶ azelaic, oxalic and valeric acids.

Toyama, Tsuchiya, *Chem. Abstracts*, 1935, **29**, 5294.
Farmer, van den Heuvel, *J. Chem. Soc.*, 1936, 1809.
Toyama, Uozaki, *Chem. Abstracts*, 1937, **31**, 249.

Purapurine.
See Solasonine.

Purine



$C_5H_4N_4$ MW, 120

Microscopic needles from EtOH or toluene. M.p. 216–17°. Very sol. H_2O, hot EtOH. Mod. sol. hot AcOEt, Me_2CO. Spar. sol. Et_2O, $CHCl_3$. Amphoteric. Very stable to ox. agents. Gives cryst. hydrochloride and cryst. metallic salts. Forms insol. Zn salt.

B,HNO₃: cryst. from H_2O. M.p. 205° decomp. Very sol. hot H_2O. Spar. sol. hot EtOH.

Picrate: plates from hot H_2O. M.p. 208°.

7-*Me*: $C_6H_6N_4$. MW, 134. Needles from EtOH. M.p. 184°. Insol. alkalis. Sol. H_2O, boiling EtOH. Spar. sol. C_6H_6. Heat of comb. C_v 820·6 Cal. Hydrochloride, nitrate, sulphate, very sol. H_2O. Forms cryst. Pt, Ag, Au salts. *$HgCl_2$ salt*: prisms from H_2O. M.p. 252°. *Methiodide*: yellow needles from MeOH. M.p. 231–2°. Very sol. H_2O.

9-*Me*: needles from toluene. M.p. 162–3°. Very sol. H_2O, EtOH. Sol. toluene. Sublimes.

Montequi, *Chem. Abstracts*, 1927, **21**, 3353.
Fischer, *Ber.*, 1899, **32**, 493; 1898, **31**, 2564.

Purpuric Acid

$C_8H_5O_6N_5$ MW, 267

Not known in free state. Acidification of salt sols. ⟶ uramil and alloxan.

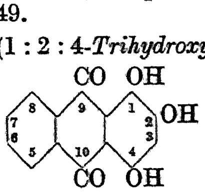
NH_4 salt: *see* Murexide.

A,CH_3·NH_2: cryst. + $1H_2O$. Anhyd. at 110°, decomp. at 210°. Mod. sol. H_2O. Spar. sol. Py. Insol. Et_2O, C_6H_6.

A,CH_3·CH_2·NH_2: red prisms + $1H_2O$. Anhyd. at 110°, decomp. at 205°.

Hantzsch, Robison, *Ber.*, 1910, **43**, 92.
Möhlau, Litter, *J. prakt. Chem.*, 1906, **73**, 449.

Purpurin (1 : 2 : 4-*Trihydroxyanthraquinone*)



$C_{14}H_8O_5$ MW, 256

As glucoside in madder root. Long orange-red or orange-yellow needles + H_2O from EtOH.Aq.; dark red needles from EtOH. Anhyd. at 100°, m.p. 259° (253°). Very sol. boiling AcOH, boiling C_6H_6, EtOH. Spar. sol. boiling H_2O. Sol. Et_2O ⟶ deep yellow fluorescent sol. Sol. CS_2. Sol. H_2SO_4 ⟶ red sol. Sol. alkalis ⟶ red sols. Ox. ⟶ phthalic acid. H_2SO_4 ⟶ purpurin-3-sulphonic acid. Red. ⟶ purpuroxanthin. Br ⟶ 3-bromo deriv. NH_3 ⟶ 4-aminopurpuroxanthin. Gives cryst. Na, K, Pb salts.

2-*Me ether*: $C_{15}H_{10}O_5$. MW, 270. Brick-red needles from C_6H_6. M.p. 232–3° (240–2°). Insol. carbonate sols. Sol. alkalis ⟶ bluish-red col. Very sol. EtOH, C_6H_6. 1-*Acetyl*: yellow needles from Me_2CO. M.p. 224–5°. 1 : 4-*Diacetyl*: yellow needles. M.p. 170–2°.

2 : 4-*Di-Me ether*: $C_{16}H_{12}O_5$. MW, 284. Orange needles. M.p. 186–9°. *Acetyl*: lemon-yellow needles from EtOH. M.p. 189–90°.

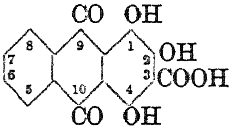
2-*Acetyl*: orange needles from EtOH. M.p. 179–80°. Sol. hot C_6H_6, hot AcOH. Spar. sol. EtOH to fluorescent sol.

2 : 4-*Diacetyl*: m.p. 175–8°. $FeCl_3$ ⟶ iodine-brown col.

Triacetyl: pale yellow needles. Sinters at 193°, m.p. 198–200°.

du Pont, U.S.P., 1,985,452, (*Chem. Zentr.*, 1935, II, 439).
Newport Chem. Corp., U.S.P., 1,790,932, (*Chem. Abstracts*, 1931, **25**, 1539).
Marshall, *J. Chem. Soc.*, 1931, 3206.
Perkin, Storey, *J. Chem. Soc.*, 1928, 238.

Purpurin-3-carboxylic Acid (*Pseudopurpurin*, 1 : 3 : 4-*trihydroxyanthraquinone*-2-*carboxylic acid*)



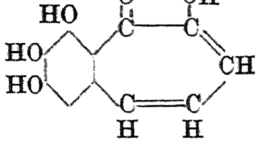
$C_{15}H_8O_7$ MW, 300

In madder root. Cryst. M.p. 222–4° decomp. Sol. alkalis, H_2SO_4. Mod. sol. boiling C_6H_6, $CHCl_3$. Sol. hot H_2O ⟶ orange-red sol. Insol. cold H_2O, EtOH. At 180–95° ⟶ purpurin. Ac_2O at 180° ⟶ triacetylpurpurin.

Baeyer, D.R.P., 272,301, (*Chem. Zentr.*, 1914, I, 1474).
Liebermann, Plath, *Ber.*, 1877, **10**, 1618.

Purpurin-8-carboxylic Acid.
See Boletol.

Purpurogallin



$C_{11}H_8O_5$ MW, 220

In gall of *Dryophanta divisa* as glycoside. Yellow or dark red needles from AcOH. M.p. 274° (rapid heat.). Decomp. without melting when slowly heated. Sol. Et_2O, $CHCl_3$. Mod. sol. EtOH. Spar. sol. H_2O. Reduces Au, Ag and alk. Cu sols. Characteristic col. with alkalis, orange-red ⟶ blue ⟶ green ⟶ dark yellow. H_2SO_4 ⟶ orange-red col. Gives spar. sol. Ba salt.

Tetra-acetyl: colourless plates from EtOH or C_6H_6. M.p. 182–3°. Sol. EtOH, Et_2O. Insol. H_2O.

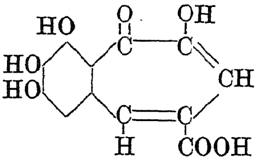
Tribenzoyl: colourless prisms. M.p. 212–13°. Insol. EtOH.

Tri-Me ether: $C_{14}H_{14}O_5$. MW, 262. Orange needles from AcOEt. M.p. 174–7°. Spar. sol. EtOH. Insol. aq. alkalis. *Acetyl*: needles from EtOH. M.p. 140–3°. Spar. sol. EtOH.

Tetra-Me ether: $C_{15}H_{16}O_5$. MW, 276. Prisms from EtOH. M.p. 93–4°. Sol. EtOH.

Barltrop, Nicholson, *J. Chem. Soc.*, 1948, 116.
Haworth, Moore, Pauson, *J. Chem. Soc.*, 1948, 1045; 1949, 3271.
Caunt, Crow, Haworth, Vodoz, *J. Chem. Soc.*, 1950, 1631.
Critchlow, Haworth, Pauson, *J. Chem. Soc.*, 1951, 1318.

Purpurogallin-carboxylic Acid



$C_{12}H_8O_7$ MW, 264

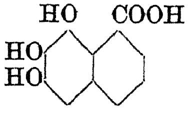
Orange microcryst. powder. Decomp. at 320°. Spar. sol. $PhNO_2$, EtOH.

Me ester: dark red needles from Me_2CO. M.p. 267–8°.

Tetra-Me ether: pale yellow needles from dioxan-pet. ether. M.p. 185–6°. *Me ester*: colourless needles from EtOH. M.p. 120–1°.

Perkin, Perkin, *J. Chem. Soc.*, 1908, **93**, 1186.
Crow, Haworth, *J. Chem. Soc.*, 1951, 1325.

Purpurogallone (6 : 7 : 8 - *Trihydroxy* - 1 - *naphthoic acid*)



$C_{11}H_8O_5$ MW, 220

Obtained by action of KOH on purpurogallin. Lemon-yellow needles. M.p. 271–3°. Sol. EtOH, AcOH, $PhNO_2$.

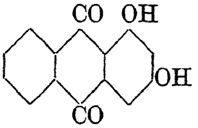
Acetyl anhydro: m.p. 174–6°.

Tri-Me ether: colourless plates from C_6H_6–cyclohexane. M.p. 145–6°. *Me ester*: yellow leaflets. M.p. 81–3°.

Et ester: m.p. 151–3°. *Acetyl*: m.p. 126–7°.

Perkin, Steven, *J. Chem. Soc.*, 1903, **83**, 192.
Perkin, *J. Chem. Soc.*, 1912, **101**, 808.
Haworth, Moore, Pauson, *J. Chem. Soc.*, 1949, 3271.

Purpuroxanthin (1 : 3-*Dihydroxyanthraquinone, xanthopurpurin*)



$C_{14}H_8O_4$ MW, 240

Yellow needles or leaflets. M.p. 268–70° (264°). Sol. Me_2CO, AcOH, $PhNO_2$. Mod. sol. EtOH, C_6H_6. Sol. alkalis ⟶ red. sols. Sublimes. Alk. fusion or $H_2SO_4 + MnO_2$ ⟶ purpurin. Zn dust dist. ⟶ anthracene. HNO_3 ⟶ phthalic acid.

1-*Acetyl*: orange-yellow needles from MeOH. M.p. 231–5°. Very sol. Me_2CO.

3-*Acetyl*: yellow needles from EtOH. M.p. 144°.

Diacetyl: pale yellow needles. M.p. 183–4°.

1-*Me ether*: $C_{15}H_{10}O_4$. MW, 254. Yellow leaflets from Me_2CO. M.p. 311–13°. *Acetyl*: yellow leaflets from EtOH. M.p. 154–5°.

3-*Me ether*: pale yellow needles from AcOH. M.p. 193°.

Di-Me ether: $C_{16}H_{12}O_4$. MW, 268. Yellow needles. M.p. 151–3°.

Di-phenyl ether: 1 : 3-diphenoxyanthraquinone. Yellow needles from AcOH. M.p. 167°.

Perkin, Story, *J. Chem. Soc.*, 1929, 1399.

Purpuroxanthin-2-carboxylic Acid.
See Munjistin.

Putrescine (*Tetramethylenediamine*, 1 : 4-*diaminobutane*)

$H_2N{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2$

$C_4H_{12}N_2$ MW, 88

Product of decomp. of excreta, dead animal matter, etc. Cryst. with strong odour. M.p. 27–8° (24°). B.p. 158–9°. D_4^{25} 0·877. Sol. H_2O. Absorbs CO_2. $k = 5{\cdot}1 \times 10^{-4}$ at 25°. Salts are not poisonous.

B,2HCl: needles from H_2O. Does not melt below 290°. Very sol. H_2O. Insol. MeOH.

B,2HAuCl$_4$: cryst. + $2H_2O$. M.p. 235–40°.

B,H$_2$*PtCl*$_6$: darkens at 230°. Does not melt below 275°. Spar. sol. cold H_2O. Insol. EtOH.

N : N′-*Diacetyl*: m.p. 137°. Sol. H_2O, EtOH.

Monobenzoyl: colourless viscous oil. B.p. 176–8°/0·1 mm. *Hydrochloride*: m.p. 167°. N′-*Acetyl*: m.p. 143°. *Picrate*: m.p. 168–70°.

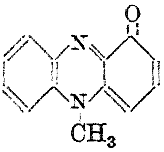
N : N′-*Dibenzoyl*: m.p. 177°.

N : N′-*Di*-m-*nitrobenzoyl*: m.p. 246°.

Di-p-toluenesulphonyl: m.p. 224°.
Picrate: pale yellow prisms. Turns brown at 250°. Decomp. at 250–5°.
N : N′-*Di-Me*: $C_6H_{16}N_2$. MW, 116. B.p. 168°. *Di-m-nitrobenzoyl*: cryst. from MeOH. M.p. 118°.
N : N′-*Tetra-Me*: $C_8H_{20}N_2$. MW, 144. Colourless liq. B.p. 168°. Misc. with hot H_2O. Sol. EtOH, Et_2O. $D_4^{18.9}$ 0·8041. $n_a^{18.9}$ 1·4316. Volatile in steam. *B,2HCl*: prisms from EtOH. M.p. 273° decomp. *B,2HAuCl*$_4$: yellow prisms from hot H_2O. Sinters at 200°. Decomp. at 206–7°. *B,H*$_2$*PtCl*$_6$: prisms + $2H_2O$. M.p. 234° decomp. *Picrate*: m.p. 198–9°.

Braun, Pirkernelle, *Ber.*, 1934, **67**, 1056.
Keil, *Z. physiol. Chem.*, 1931, **196**, 81.
Wrede, Fanselow, Strack, *Z. physiol. Chem.*, 1927, **163**, 219.
Keil, *Ber.*, 1926, **59**, 2816.
Chambret, Joly, *Bull. soc. chim.*, 1947, 1023.
Schultz, *J. Am. Chem. Soc.*, 1948, **70**, 2666.

Pyocyanine

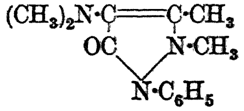


$C_{13}H_{10}ON_2$ MW, 210

Pigment of *Bacillus pyocyaneus*. Dimer. Blue needles from $CHCl_3$–pet. ether. M.p. 133°. Sol. $CHCl_3$, Me_2CO, AcOEt, hot H_2O. Insol. Et_2O, C_6H_6, pet. ether, CCl_4, CS_2. Hyd. ⟶ 2 mols. hemipyocyanine. Acetylation or benzoylation ⟶ hemipyocyanine acetate or benzoate. Increases respiration of living cells.
Perchlorate: red needles. M.p. 221–3° decomp.
Picrate: red leaflets + 2MeOH from MeOH, or violet leaflets + 1EtOH from EtOH. M.p. 194–5° decomp.
Picrolonate: reddish-black cryst. + 1EtOH. M.p. 195–6°.

Wrede, Strack, *Z. physiol. Chem.*, 1929, **181**, 58; *Ber.*, 1929, **62**, 2051.
McCombie, Scarborough, *J. Chem. Soc.*, 1923, **123**, 3279.
Surrey, *Organic Syntheses*, 1946, XXVI, 86.

Pyramidone (*4-Dimethylaminoantipyrine, amidopyrine*)



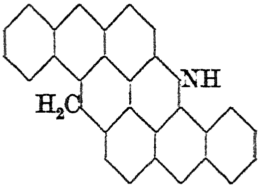
$C_{13}H_{17}ON_3$ MW, 231

Cryst. powder. M.p. 108°. Sol. H_2O, EtOH, Et_2O, C_6H_6. $FeCl_3$ ⟶ bluish-violet col. Antipyretic. Less toxic and more powerful than antipyrine.

B,HBr: pyrobromone. Needles. M.p. 190°. Sol. H_2O, EtOH. Insol. Et_2O, C_6H_6.
B,HI: pyro-iodone. M.p. 196–7°.
B, β-Camphorsulphonic acid: m.p. 180° decomp. $[\alpha]_D^{20}$ + 10·4°.
Citrate: cryst. from EtOH.Aq. M.p. 85°.
B,HAu(CN)$_4$: plates. M.p. 183–5°.
Methobromide: decomp. at 212°.
Methiodide: decomp. about 220°. Sol. H_2O.
Picrate: m.p. 168–70°.
Styphnate: yellow needles from EtOH. M.p. 191°.

Rodionov, *Bull. soc. chim.*, 1926, **39**, 305.
Klebanskiĭ, Lemke, *Chem. Abstracts*, 1935, **29**, 6891.
Reuter, U.S.P., 2,005,506, (*Chem. Abstracts*, 1935, **29**, 5130).
I.G., D.R.P., 617,237, (*Chem. Zentr.*, 1935, II, 3949).
Knorr, Stolz, *Ann.*, 1896, **293**, 66.

Pyranthridine

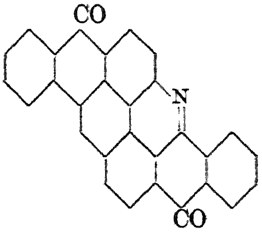


$C_{29}H_{17}N$ MW, 379

Reddish-brown leaflets from xylene. M.p. 370°. Conc. H_2SO ⟶ dark blue salt.

Scholl, Dischendorfer, *Ber.*, 1918, **51**, 441.

Pyranthridone

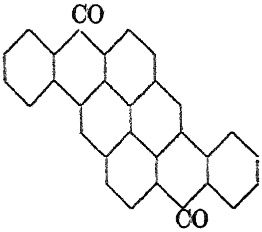


$C_{29}H_{13}O_2N$ MW, 407

Brownish-yellow needles from quinoline. Does not melt below 500°. Sublimes. Sol. conc. H_2SO_4 with red col. Insol. most org. solvents. Gives violet-blue vat.

Scholl, Dischendorfer, *Ber.*, 1918, **51**, 441.
Scholl, D.R.P., 307,390, (*Chem. Zentr.*, 1918, II, 495).

Pyranthrone



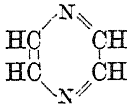
$C_{30}H_{14}O_2$ MW, 406

Brown cryst. Sol. hot $PhNO_2$, hot aniline. Sol. conc. H_2SO_4 with blue col. Well known orange vat dyestuff.

Scholl, *Ber.*, 1910, **43**, 350; D.R.P., 239,761, (*Chem. Zentr.*, 1911, II, 1498).
Badische, D.R.P., 175,067, (*Chem. Zentr.*, 1906, II, 1537).
Gallotti, *Chem. Abstracts*, 1934, **28**, 5063.
Lecher, Scalera, Forster, U.S.P., 2,416,931, (*Chem. Abstracts*, 1947, **41**, 5152).
Lee, Deinet, Gottlieb, U.S.P., 2,405,702, (*Chem. Abstracts*, 1947, **41**, 157).

Pyrantin.
See under Succinimide.

Pyrazine (1 : 4-*Diazine*)

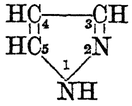


$C_4H_4N_2$ MW, 80

Prisms from H_2O. M.p. 47°. B.p. 115·5–115·8°/768·4 mm. $D_4^{60·9}$ 1·0311. $n_D^{60·9}$ 1·4953. Sol. H_2O, EtOH, Et_2O. Volatile in steam.
B,H₂SO₄ : m.p. 136–7°.
B,HAuCl₄ : yellow plates from HCl. M.p. 245° decomp.
B,AuCl₃ : m.p. 202–3°.
B,HgCl₂ : m.p. 273° decomp.
Picrate : yellow needles from EtOH. M.p. 157°.

Aston, Peterson, Holowchzk, *J. Am. Chem. Soc.*, 1934, **56**, 153.
Wolff, Marburg, *Ann.*, 1906, **363**, 215.
Stoehr, *J. prakt. Chem.*, 1894, **49**, 392; 1895, **51**, 449.
Gabriel, Pinkus, *Ber.*, 1893, **26**, 2207.

Pyrazole (1 : 2-*Diazole*)

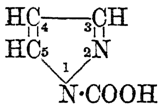


$C_3H_4N_2$ MW, 68

Needles or prisms from ligroin. M.p. 69·5–70°. B.p. 186–8°/757·9 mm. Sol. H_2O, EtOH, Et_2O, C_6H_6. $k = 3·0 \times 10^{-12}$ at 25°.
B,HCl : hygroscopic needles. M.p. 104°.
B₂,H₂SO₄ : cryst. from EtOH. M.p. 134°.
B,HNO₃ : needles. M.p. 148°.
B,(COOH)₂ : needles from EtOH–Et_2O. M.p. 192° decomp.
N-*Me* : *see* 1-Methylpyrazole.
N-*Phenyl* : *see* 1-Phenylpyrazole.
N-*Acetyl* : oil. B.p. 155–6°/744 mm. *Picrate* : yellow prisms from EtOH. M.p. 170–1°.
N-*Benzoyl* : m.p. 46°. B.p. 281°/747 mm., 220–5°/60 mm., 140°/11 mm.
Picrate : yellow needles. M.p. 160°.

v. Pechmann, *Ber.*, 1898, **31**, 2950.
Curtius, Wirsing, *J. prakt. Chem.*, 1894, **50**, 544.
Dains, Harger, *J. Am. Chem. Soc.*, 1918, **40**, 562.
Mingoia, *Chem. Zentr.*, 1931, II, 2324.
Knorr, D.R.P., 74,619.
Hückel, Datow, Simmersbach, *Z. physik. Chem.*, 1940, *A***186**, 129.

Pyrazole-1-carboxylic Acid



$C_4H_4O_2N_2$ MW, 112

M.p. 102–3° decomp. Boil with $H_2O \longrightarrow$ pyrazole.
Et ester : $C_6H_8O_2N_2$. MW, 140. Oil. B.p. 213°/741 mm., 120–3°/42 mm.
Amide : $C_4H_5ON_3$. MW, 111. Cryst. from EtOH. M.p. 136·5°.

Mingoia, *Chem. Zentr.*, 1931, II, 2324.
Knorr, *Ber.*, 1895, **28**, 716.

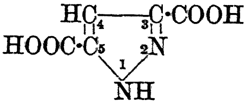
Pyrazole-3-carboxylic Acid.
Prisms from H_2O. M.p. 210–12°. Sol. H_2O, EtOH. Mod. sol. Et_2O, AcOH. Insol. $CHCl_3$, C_6H_6.
Me ester : $C_5H_6O_2N_2$. MW, 126. Cryst. from C_6H_6. M.p. 139–40°. Sol. MeOH. Spar. sol. ligroin.
N-*Me* : $C_5H_6O_2N_2$. MW, 126. Cryst. M.p. 222°. Mod. sol. hot H_2O.
N-*Phenyl* : *see* 1-Phenylpyrazole-3-carboxylic Acid.

Auwers, Breyhan, *J. prakt. Chem.*, 1935, **143**, 274.
Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 187.
v. Pechmann, Burkhard, *Ber.*, 1900, **33**, 3595.
Buchner, Kachumian, *Ber.*, 1902, **35**, 41.
Knorr, Macdonald, *Ann.*, 1894, **279**, 231.
Jowett, Potter, *J. Chem. Soc.*, 1903, **83**, 469.

Pyrazole-4-carboxylic Acid.
Needles from H_2O. M.p. 219–20° (decomp. at 275°). Sol. EtOH. Mod. sol. H_2O, Et_2O. Sublimes.
N-*Phenyl* : *see* 1-Phenylpyrazole-4-carboxylic Acid.

Knorr, Laubmann, *Ber.*, 1889, **22**, 180.
Behagel, Buchner, *Ber.*, 1902, **35**, 35.

Pyrazole-3 : 5-dicarboxylic Acid



$C_5H_4O_4N_2$ MW, 156

Needles. M.p. 287–90° decomp. $D^{22.5}$ 1·626. Spar. sol. H_2O, EtOH, Et_2O. Very spar. sol. $CHCl_3$, ligroin.

Di-Me ester: $C_7H_8O_4N_2$. MW, 184. Plates from Et_2O. M.p. 151·5°. Sol. EtOH. Spar. sol. ligroin. N-*Me*: needles from ligroin. M.p. 72–73·5°. N-*Acetyl*: plates from EtOH. M.p. 84·5–85°.

Chloride: m.p. 86–7°.

Anhydride: m.p. 310°.

Dimorpholide: m.p. 194–6° (184°).

N-*Phenyl*: *see* 1-Phenylpyrazole-3 : 5-dicarboxylic Acid.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 196.
Gray, *Ber.*, 1900, **33**, 1223.
Knorr, Macdonald, *Ann.*, 1894, **279**, 218 (*Footnote*).

Pyrazole-4 : 5-dicarboxylic Acid.

Needles + $1H_2O$ from dil. HNO_3. M.p. 260° decomp. Sol. EtOH. Spar. sol. Et_2O. Insol. $CHCl_3$. Careful heating ⟶ pyrazole-4-carboxylic Acid.

Di-Me ester: needles from H_2O. M.p. 141°.

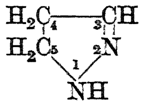
Dianilide: m.p. 244°.

N-*Acetyl*: m.p. 169°.

N-*Phenyl*: *see* 1-Phenylpyrazole-4 : 5-dicarboxylic Acid.

v. Pechmann, *Ber.*, 1900, **33**, 630.
v. Pechmann, Sell, *Ber.*, 1899, **32**, 2299.

2-Pyrazoline (4 : 5-*Dihydropyrazole*)



$C_3H_6N_2$ MW, 70

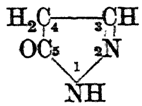
Colourless liq. B.p. 144°. D_4^{20} 1·017. n_{He}^{20} 1·478. Misc. with H_2O, EtOH. Volatile in steam and ether.

N-o-*Tolyl*: liq. B.p. 271°/759·5 mm. D_0^0 1·084.

N-p-*Tolyl*: needles from EtOH.Aq. M.p. 60·5°. B.p. 281–2°/758 mm.

Auwers, Heimke, *Ann.*, 1927, **458**, 180.

5-Pyrazolone



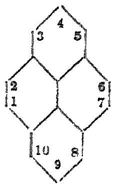
$C_3H_4ON_2$ MW, 84

Needles from toluene. M.p. 165°. Sol. H_2O, EtOH. Spar. sol. Et_2O.

4-*Isonitroso*: needles from H_2O. M.p. 180–1°.

Ruhemann, Morrel, *Ber.*, 1895, **28**, 988.
Knorr, *Ber.*, 1896, **29**, 253.
Dains, O'Brien, Johnson, *J. Am. Chem. Soc.*, 1916, **38**, 1510.

Pyrene



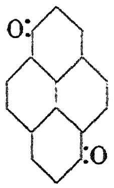
$C_{16}H_{10}$ MW, 202

Constituent of coal tar. Pale yellow plates by cryst. or sublimation. M.p. 149–50°. Does not boil below 360°. Sol. Et_2O, CS_2, C_6H_6, toluene. Sols. show blue fluor. CrO_3 ⟶ pyrenequinone ⟶ pyrenic acid.

Picrate: red needles from EtOH. M.p. 222°. Mod. sol. hot EtOH. Sol. CS_2, C_6H_6.

Clar, *Ber.*, 1936, **69**, 1683.
Fleischer, Retze, *Ber.*, 1922, **55**, 3280.
Weitzenböck, *Monatsh.*, 1913, **34**, 221.
Graebe, *Ann.*, 1871, **158**, 285.
Baker, McOmie, Norman, *Chemistry and Industry*, 1950, 77.

Pyrene-3 : 8-quinone

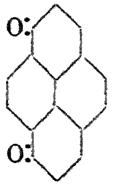


$C_{16}H_8O_2$ MW, 232

Yellow needles from $PhNO_2$. M.p. 309° (rapid heat.); darkens at 290° (slow heat.). Sol. conc. H_2SO_4 with yellowish-orange col. Sol. $PhNO_2$. Spar. sol. EtOH, Et_2O, CS_2, C_6H_6.

Vollmann, Becker, Corell, Streeck, *Ann.*, 1937, **531**, 1.
Goldschmiedt, *Monatsh.*, 1883, **4**, 310.
Griebe, *Ann.*, 1871, **158**, 295.
See also Scholl, Seer, *Ann.*, 1912, **394**, 125.

Pyrene-3 : 10-quinone

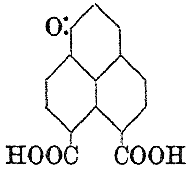


$C_{16}H_8O_2$ MW, 232

Brownish-red needles from AcOH. M.p. 270°. Sol. conc. H_2SO_4 with olive-green col.

Vollmann, Becker, Corell, Streeck, *Ann.*, 1937, **531**, 1.

Pyrenic Acid



$C_{15}H_8O_5$ MW, 268

Pale yellow plates.

α-Me ester: $C_{16}H_{10}O_5$. MW, 282. Yellow needles from MeOH. Decomp. at 275°. Sol. boiling EtOH to 0·5%.

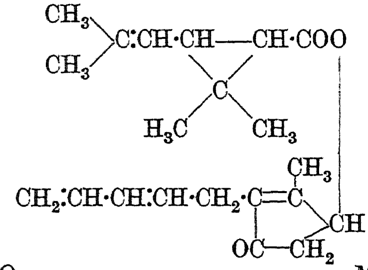
β-Me ester: yellowish-green needles from MeOH. Sol. boiling EtOH to 1·06%.

Anhydride: $C_{15}H_6O_4$. MW, 250. Needles. Decomp. at 260°.

Imide: $C_{15}H_{11}O_2N$. MW, 237. Yellow plates. Insol. cold alkalis, sol. hot alkalis ⟶ red sols.

Bamberger, Philip, *Ann.*, 1887, **240**, 168.
Langstein, *Monatsh.*, 1910, **31**, 863.

Pyrethrin I



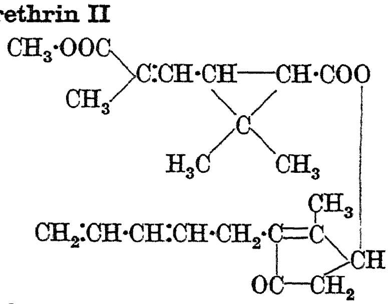
$C_{21}H_{28}O_3$ MW, 328

Insecticidal constituent of pyrethrum flowers (*Chrysanthemum cinerariifolium*). Oil. $[\alpha]_D^{25}$ − 26°. n_D^{20} 1·5192.

Semicarbazone: m.p. 114–16° (*ex* pyrethrum flowers), 75–80° (synthetic).

Harper, *Annual Reports on the Progress of Chemistry*, (London), 1948, XLV, 162.
Crombie, Harper, Thompson, *J. Chem. Soc.*, 1951, 2906.

Pyrethrin II



$C_{22}H_{28}O_5$ MW, 372

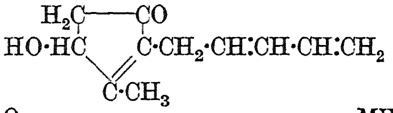
Insecticidal constituent of pyrethrum flowers (*Chrysanthemum cinerariifolium*). Oil. $[\alpha]_D^{20}$ − 6·0° in Et_2O. n_D^{20} 1·5258.

Semicarbazone: m.p. 163–4°.

Tetrahydro: $n_D^{23\cdot5}$ 1·5115. *Semicarbazone*: m.p. 139–41°.

Harper, *Annual Reports on the Progress of Chemistry*, (London), 1948, XLV, 162.

Pyrethrolone



$C_{11}H_{14}O_2$ MW, 178

Pyrethrolone-B1 (*Cis*?).

Oil. B.p. 135–7°/0·7 mm. n_D^{20} 1·5402, n_D^{25} 1·5424. $[\alpha]_D$ +14·6°, +11·7° in EtOH.

Semicarbazone: m.p. 219° (213–5°). $[\alpha]_D$ −182° in Py. *Acetyl*: needles from Et_2O or C_6H_6. M.p. 130–3° (122–4°). $[\alpha]_D^{25}$ + 49·0° in MeOH.

Tetrahydro: n_D^{26} 1·4905. $[\alpha]_D^{26}$ + 13·5° in EtOH. *Semicarbazone*: m.p. 198°. $[\alpha]_D$ − 196° in Py.

Pyrethrolone-B2 (*Trans*?).

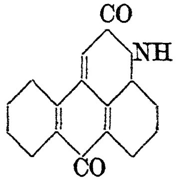
Oil. B.p. 140–2°/0·9 mm. n_D^{20} 1·5505. $[\alpha]_D$ +11·4°.

Semicarbazone: m.p. 219–20° decomp. $[\alpha]_D$ − 155° in Py. *Acetyl*: cryst. from Et_2O. M.p. 128–30°. $[\alpha]_D$ − 47·8° in MeOH, − 32·6° in Py.

Tetrahydro: *semicarbazone*, m.p. 195–7°. $[\alpha]_D$ − 207° in Py.

Staudinger, Ruzicka, *Helv. Chim. Acta*, 1924, **7**, 215.
LaForge, Barthel, *J. Org. Chem.*, 1945, **10**, 106, 114.
West, *J. Chem. Soc.*, 1946, 463.
Crombie, Harper, Thompson, *J. Chem. Soc.*, 1951, 2906.

Pyridanthrone (*Anthrapyridone*)



$C_{16}H_9O_2N$ MW, 247

Cryst. from $PhNO_2$. M.p. 406–8°. Sol. conc. H_2SO_4 with yellow col. and yellow fluor.

N-*Me*: $C_{17}H_{11}O_2N$. MW, 261. Cryst. from $PhNO_2$. M.p. 267–8°.

Seka, Schreckental, Heilperin, *Monatsh.*, 1929, **53** and **54**, 478.
Höchst, D.R.P., 250,885, (*Chem. Zentr.*, 1912, II, 1319).
Bayer, D.R.P., 209,033, (*Chem. Zentr.*, 1909, I, 1680).
Badische, D.R.P., 216,597, (*Chem. Zentr.*, 1910, I, 68).

Pyridazine (1 : 2-*Diazine, orthodiazine*)

$C_4H_4N_2$ MW, 80

Liq. F.p. — 8°. B.p. 208°, 47–8°/1 mm. $D_4^{23.5}$ 1·1035. $n_D^{23.5}$ 1·5231. Very sol. H_2O. Sol. EtOH, Et_2O, C_6H_6. Insol. pet. ether. Na + EtOH ⟶ putrescine.

B,AuCl₃: yellow needles from H_2O. M.p. 170° decomp. Insol. cold H_2O.

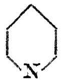
Chloroaurate: yellow cryst. M.p. 110°.

B_2,H_2PtCl_6: orange prisms. Decomp. at 218°. Sol. H_2O.

Picrate: yellow needles from H_2O or EtOH. M.p. 169° decomp.

Wohl, Bernreuther, *Ann.*, 1930, **481**, 12.
Gabriel, *Ber.*, 1909, **42**, 658.
Evans, Wiselogle, *J. Am. Chem. Soc.*, 1945, **67**, 60.

Pyridine



C_5H_5N MW, 79

Occurs in coffee oil. Extracted in quantity from coal tar. Colourless liq. with characteristic odour. F.p. —42°. B.p. 115·5°. Forms azeotropic mixture with 3 mols. H_2O, b.p. 92–3°. Very hygroscopic. Misc. with H_2O in all proportions. Volatile in steam. D_4^{25} 0·97796. n_D^{21} 1·5092. $k = 2{\cdot}4 \times 10^{-9}$ at 25°. Heat of comb. C_v 664·7 Cal., C_p 675·1 Cal. Latent heat of vap. 101·4 Cal. per kilogram at 115·5°. Sp. heat (21°) 0·391. Crit. temp. about 344°. F.p. depression 4·97° per kilogram. B.p. elevation 2·69° per kilogram. Red heat ⟶ 2 : 4′-dipyridyl. Red. ⟶ piperidine. H_2SO_4 ⟶ 3-sulphonic acid. $NaNH_2$ ⟶ 2-aminopyridine. MeOH–HCl ⟶ pyridine methochloride. $ClCH_2{\cdot}COOEt$ ⟶ pyridine-betaine hydrochloride. Used for denaturing industrial spirit. Powerful solvent.

B,HCl: hygroscopic plates from EtOH. M.p. 82°. B.p. 218–19°. Sol. H_2O, EtOH, $CHCl_3$. Insol. Et_2O.

B,2HCl: prisms. M.p. 46–7°.

B,HBr: reddish-yellow plates. M.p. 213°. Sol. H_2O, EtOH, $CHCl_3$. Insol. Et_2O.

B,HI: cryst. + $1H_2O$ from H_2O. M.p. 268° decomp.

$B,HClO_3$: cryst. from EtOH. M.p. 147° decomp.

$B,(COOH)_2$: m.p. 151–2°.

Phthalate: cryst. M.p. 109°.

Tartrate: m.p. 154°.

Citrate: m.p. 123°.

3 : 5-*Dinitrobenzoate*: yellow needles. M.p. 162°.

3 : 5-*Dinitro-o-toluate*: m.p. 143–4°.

3 : 5-*Dinitro-p-toluate*: m.p. 168–70°.

$B,HAuCl_4$: yellow needles from H_2O or EtOH. M.p. 329° (304°). Spar. sol. hot H_2O.

B,H_2PtCl_6: reddish-yellow needles. M.p. 262–4° decomp. Very sol. H_2O. Insol. EtOH.

$B_2,HgCl_2$: needles from Py. M.p. 108°.

$B,HgCl_2$: needles from H_2O or EtOH. M.p. 180°.

Methiodide: needles or prisms from EtOH or Me_2CO. Very hygroscopic. Yellows at 100°. M.p. 118°. Very sol. H_2O. Sol. EtOH, $CHCl_3$, AcOH, Me_2CO. Insol. Et_2O, C_6H_6, CS_2.

Ethobromide: cryst. from EtOH. M.p. 111–12°. Sol. H_2O, EtOH. Insol. Et_2O.

Ethiodide: plates from EtOH–Et_2O. M.p. 90·5°. Sol. H_2O, EtOH, AcOH, Me_2CO. Insol. Et_2O, $CHCl_3$, C_6H_6, CS_2.

Propiodide: plates from EtOH. M.p. 52–3°. Sol. H_2O, EtOH, AcOEt, C_6H_6. Insol. Et_2O, $CHCl_3$.

Isopropiodide: cryst. from EtOH. M.p. 114–15°. Sol. H_2O, EtOH, AcOEt. Insol. Et_2O.

Phenochloride: needles. M.p. 105–6°. Very sol. H_2O. Sol. EtOH. Insol. Et_2O.

Phenobromide: m.p. 155°.

Pheniodide: pale yellow cryst. M.p. 207°.

Picrate: yellow needles. M.p. 165–6°.

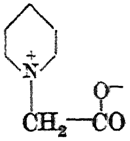
Styphnate: cryst. from EtOH. M.p. 184·5–185·5°.

Ma, Hsia, Sah, *Chem. Abstracts*, 1934, **28**, 3692.
Ferns, Lapworth, *J. Chem. Soc.*, 1912, **101**, 283.
Decker, Kaufmann, *J. prakt. Chem.*, 1911, **84**, 436.

Pyridine Aldehyde.

See Picolinic Aldehyde, Nicotinic Aldehyde *and* Isonicotinic Aldehyde.

Pyridine-betaine (*Anhydride or lactone of* N-*carboxymethylpyridinium hydroxide*)



$C_7H_7O_2N$ MW, 137

Hygroscopic plates + $1H_2O$. Anhyd. at 100°. M.p. 150° decomp. Very sol. H_2O, hot EtOH. Insol. Et_2O.

B_2,HCl,H_2O: prisms from EtOH. M.p. 159° decomp. Very sol. H_2O. Insol. EtOH, Et_2O.

B,HCl: plates from H_2O or EtOH. M.p. 202–5°. Very sol. H_2O. Sol. hot EtOH. Spar. sol. cold EtOH. Insol. Et_2O.

B_2,HBr,H_2O: plates or prisms from EtOH. Decomp. at 170°.

B,HBr: plates from EtOH.Aq. Decomp. at 198–200°.

B_2,HI: needles from EtOH. Blackens at

175–80°. M.p. 250–2° decomp. Very sol. H_2O. Sol. warm EtOH. Insol. Et_2O.

B_2,H_2SO_4: plates. Decomp. at 175°.

B,HNO_3: plates from EtOH.Aq. M.p. 145° decomp.

B_2,H_2PtCl_6: orange-red cryst. M.p. 215°. Sol. H_2O. Insol. EtOH.

Chloroaurate: m.p. 165°.

$B,AgNO_3$: plates. Decomp. at 171·5°.

$B_2,(HgCl_2)_4,HCl$: plates + $1H_2O$ from H_2O. M.p. 134°.

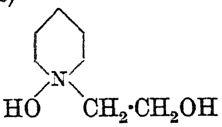
Picrate: yellow prisms from EtOH.Aq. M.p. 142–3°.

Chattaway, Garton, *J. Chem. Soc.*, 1924, **125**, 187.
Krüger, *J. prakt. Chem.*, 1891, **43**, 287.
Vongerichten, *Ber.*, 1882, **15**, 1251.

Pyridine-carboxylic Acid.

See Picolinic Acid, Nicotinic Acid *and* Isonicotinic Acid.

Pyridine-choline (N-β-*Hydroxyethylpyridinium hydroxide*)



$C_7H_{11}O_2N$ MW, 141

Chloride: β-hydroxyethylpyridinium chloride. $C_7H_{10}ONCl$. MW, 159·5. Hygroscopic prisms from EtOH. Very sol. H_2O, EtOH. Insol. Et_2O. $Ag_2O \longrightarrow$ free base. $CrO_3 \longrightarrow$ pyridine-betaine hydrochloride. *$B,AuCl_3$*: needles or plates. M.p. 117°. Very sol. boiling H_2O. *$B_2,PtCl_4$*: orange-yellow plates from EtOH. M.p. 179°. Very sol. H_2O. Insol. cold EtOH.

Schmidt, *Arch. Pharm.*, 1913, **251**, 205.

Pyridine-2 : 3-dicarboxylic Acid.
See Quinolinic Acid.

Pyridine-2 : 4-dicarboxylic Acid.
See Lutidinic Acid.

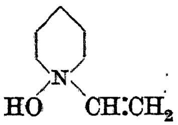
Pyridine-2 : 5-dicarboxylic Acid.
See Isocinchomeronic Acid.

Pyridine-2 : 6-dicarboxylic Acid.
See Dipicolinic Acid.

Pyridine-3 : 4-dicarboxylic Acid.
See Cinchomeronic Acid.

Pyridine-3 : 5-dicarboxylic Acid.
See Dinicotinic Acid.

Pyridine-neurine (N-*Vinylpyridinium hydroxide*)

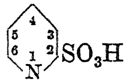


C_7H_9ON MW, 123

Chloride: vinylpyridinium chloride. C_7H_8NCl. MW, 141·5. *$B,AuCl_3$*: yellow needles from H_2O. M.p. 178°. *$B_2,PtCl_4$*: yellowish-red plates from H_2O. M.p. 193° decomp.

Schmidt, *Arch. Pharm.*, 1913, **251**, 206.

Pyridine-2-sulphonic Acid



$C_5H_5O_3NS$ MW, 159

Needles from EtOH. M.p. 247–8°. Very sol. H_2O.

NH_4 salt: m.p. 274–5°.

Ag salt: yellow cryst. from H_2O. M.p. 290° decomp. Very sol. hot H_2O.

Plazek, Marcinków, *Chem. Abstracts*, 1935, **29**, 2535.
Gastel, Wibaut, *Rec. trav. chim.*, 1934, **53**, 1031.
Marckwald, Klemm, Trabert, *Ber.*, 1900, **33**, 1560.

Pyridine-3-sulphonic Acid.

Needles or plates. M.p. 357°. Very sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. Dry dist. $\longrightarrow$ 3 : 3′-dipyridyl. KOH fusion $\longrightarrow$ 3-hydroxypyridine.

NH_4 salt: cryst. M.p. 243°.

Betaine: $C_6H_7O_3NS$. MW, 173. Cryst. from H_2O. M.p. 130°.

Chloride: *hydrochloride*, m.p. 141–4° decomp.

Amide: m.p. 110–11°.

Diethylamide: m.p. 49–50°. B.p. 183–6°. *Hydrochloride*: m.p. 133–4°. *Methiodide*: m.p. 186–8°.

Anilide: m.p. 145°.

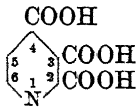
Gastel, Wibaut, *Rec. trav. chim.*, 1934, **53**, 1031.
I.G., F.P., 685,062, (*Chem. Abstracts*, 1930, **24**, 5307).
Fischer, *Ber.*, 1882, **15**, 62.
McIlvain, Goese, *J. Am. Chem. Soc.*, 1943, **65**, 2233.
Machek, *Monatsh*, 1938, **72**, 77.

Pyridine-4-sulphonic Acid.

Needles from EtOH. M.p. 134–5°.

Koenigs, Kinne, *Ber.*, 1921, **54**, 1357.
Tiesler, U.S.P. 2,330,641, (*Chem. Abstracts*, 1944, **38**, 1249).

Pyridine-2 : 3 : 4-tricarboxylic Acid (*Carbocinchomeronic acid*)



$C_8H_5O_6N$ MW, 211

Cryst. + $1\frac{1}{2}H_2O$ from H_2O. M.p. anhyd. 249–50° (rapid heat.). Sol. hot H_2O. Mod. sol. hot EtOH. Insol. Et_2O, C_6H_6. Heat at 170–80° or boiling AcOH $\longrightarrow$ cinchomeronic acid.

Tri-Me ester: $C_{11}H_{11}O_6N$. MW, 253. Needles. M.p. 102°. *Hydrochloride*: m.p. 68°.

3 : 4-*Di-Et ester*: $C_{12}H_{13}O_6N$. MW, 267. M.p. 118°. Sol. most ord. org. solvents. *Hydrochloride*: m.p. 142°.

Tri-Et ester: $C_{14}H_{17}O_6N$. MW, 295. B.p. 300–5°. *Hydrochloride*: m.p. 61°.

Mumm, Hüneke, *Ber.*, 1918, **51**, 159.
Eckert, Lorià, *Monatsh.*, 1917, **38**, 244.

Pyridine-2 : 3 : 5-tricarboxylic Acid (*Carbodinicotinic acid*).

Cryst. + $1\frac{1}{2}H_2O$ from H_2O. M.p. 323°. Sol. hot H_2O, EtOH.

Weber, *Ann.*, 1887, **241**, 11.

Pyridine-2 : 3 : 6-tricarboxylic Acid.

Needles or plates + $2H_2O$ from H_2O or EtOH.Aq. M.p. 130°. Very sol. H_2O, EtOH.Aq. Spar. sol. EtOH, Et_2O, AcOH. Heat above m.p. ⟶ pyridine-2 : 5-dicarboxylic acid. Gives cryst. spar. sol. Ca salt.

Eckert, Lorià, *Monatsh.*, 1917, **38**, 241.
Miller, *Ber.*, 1891, **24**, 1916.

Pyridine-2 : 4 : 5-tricarboxylic Acid.

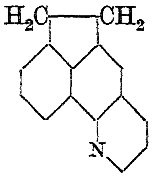
See Berberonic Acid.

Pyridine-3 : 4 : 5-tricarboxylic Acid. (β-*Carbocinchomeronic acid*).

Cryst. in leaflets + $3H_2O$ from H_2O. Chars at 261°. Sol. hot H_2O.

Weber, *Ann.*, 1887, **241**, 16.

4 : 5-[α : β′-Pyridino]-acenaphthene (*Acenaphthapyridine, acenaphthaquinoline*)



$C_{15}H_{11}N$ MW, 205

Needles from Et_2O. M.p. 67°. Sol. EtOH, Et_2O, AcOH, $CHCl_3$, C_6H_6. Sol. dil. AcOH with blue fluor. Sol. conc. H_2SO_4 with yellowish green col.

B,HCl: yellow needles from EtOH. M.p. 305°.

B,H_2SO_4: yellow needles from EtOH. M.p. 238°.

Methiodide: yellow needles from MeOH. M.p. above 315°.

Picrate: m.p. 234°.

Zinke, Raith, *Monatsh.*, 1919, **40**, 273.
Stewart, *J. Chem. Soc.*, 1925, **127**, 1331.

Pyridinoanthracene.

See Anthraquinoline.

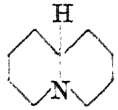
Pyridinoanthraquinone.

See Anthraquinolinequinone.

Pyridinostyrene.

See Stilbazole.

Pyridocoline (9H-*Quinolizine*)



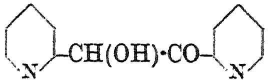
C_9H_9N MW, 131

Oil. B.p. 210–20°.

Picrate: m.p. 96°.

Diels *et al.*, *Ann.*, 1932, **498**, 16.

Pyridoin (α-*Hydroxy*-β-*keto-dipyridylethane*)



$C_{12}H_{10}O_2N_2$ MW, 214

Yellow needles from EtOH. M.p. 156°. Sol. hot EtOH, Me_2CO, AcOEt, $CHCl_3$, C_6H_6. Insol. H_2O. Dil. AcOH ⟶ red col. Reduces Fehling's.

Harries, Lénárt, *Ann.*, 1915, **410**, 108.

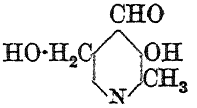
Pyridone.

See Hydroxypyridine.

Pyridopyridine.

See Naphthyridine.

Pyridoxal (3-*Hydroxy*-2-*methyl*-5-*hydroxymethyl-isonicotinic aldehyde*)



$C_8H_9O_3N$ MW, 167

Oxidation product of adermin.

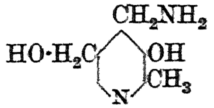
Oxime: m.p. 225–6° decomp.

Semicarbazone: m.p. 235° decomp.

O-*Phosphate*: *see* Codecarboxylase.

Harris, Heyl, Folkers, *J. Am. Chem. Soc.*, 1944, **66**, 2088.

Pyridoxamine (3-*Hydroxy*-2-*methyl*-4-*aminomethyl*-5-*hydroxymethyl-pyridine*)



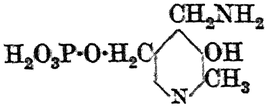
$C_8H_{12}O_2N_2$ MW, 168

Colourless cryst. M.p. 193–193·5°. Sol. EtOH, aq. acids.

B,2HCl: cryst. M.p. 226–7° decomp.

Harris, Heyl, Folkers, *J. Am. Chem. Soc.*, 1944, **66**, 2088.

Pyridoxamine phosphate



$C_8H_{13}O_5N_2P$ MW, 248

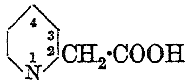
Coenzyme of transamination. Rhombic plates + $2H_2O$ from EtOH.Aq. Loses $2H_2O$

over P_2O_5. Ox. with MnO_2 ⟶ codecarboxylase.

Rabinowitz, Snell, *J. Biol. Chem.*, 1947, **169**, 643.
McNutt, Snell, *J. Biol. Chem.*, 1948, **173**, 801.
Heyl, Luz, Harris, Folkers, *J. Am. Chem. Soc.*, 1951, **73**, 3436.
Wilson, Harris, *ibid.*, 4693.
Peterson, Sober, Meister, *J. Am. Chem. Soc.*, 1952, **74**, 570.

Pyridoxin.
See Adermin.

2-Pyridylacetic Acid



$C_7H_7O_2N$ MW, 137

Cryst. M.p. 98°. Boiling aq. sol. ⟶ α-picoline.
Me ester: $C_8H_9O_2N$. MW, 151. B.p. 123°/12 mm.
Anilide: m.p. 134°.
Picrate: m.p. 142°.

Oparina, *Chem. Zentr.*, 1935, I, 2536.

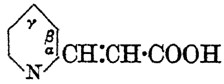
3-Pyridylacetic Acid.
M.p. 144°.
B,HCl: m.p. 153–5°.
Me ester: b.p. 112°/10 mm. *Methiodide*: m.p. 85°.
Et ester: b.p. 124°/12 mm. *Picrate*: m.p. 110–12°.
Amide: m.p. 123°.
Picrate: m.p. 99–101°.

Hartmann, Bosshard, *Helv. Chim. Acta*, 1941, **24**, 28E.
Malan, Dean, *J. Am. Chem. Soc.*, 1947, **69**, 1797.

4-Pyridylacetic Acid.
B,HCl: m.p. 130–1°.
Et ester: b.p. 107–8°/3 mm. *Picrate*: m.p. 121–3°.
Picrate: m.p. 114–16°.

Malan, Dean, *J. Am. Chem. Soc.*, 1947, **69**, 1797.

2-α-Pyridylacrylic Acid



$C_8H_7O_2N$ MW, 149

Needles from H_2O. M.p. 202–3° decomp. Very sol. EtOH. Insol. cold H_2O. Red. ⟶ 2-α-pyridylpropionic acid.
Me ester: $C_9H_9O_2N$. MW, 163. *B,HCl*: cryst. from MeOH. M.p. 185–6°.
Et ester: $C_{10}H_{11}O_2N$. MW, 177. F.p. 4°. B.p. 161°/25 mm. *B,HAuCl₄*: yellow needles. M.p. 149°. *B_2,H_2PtCl_6*: yellow needles. M.p. 114°.
B,HCl: cryst. from EtOH. M.p. 220° decomp.
B,HBr: cryst. from AcOH. M.p. 222–3°.
$B,HAuCl_4$: yellow needles from H_2O. M.p. 194–5°.
B_2,H_2PtCl_6: reddish-yellow prisms. M.p. 213° decomp.
Methobromide: cryst. from AcOH. M.p. 242°. Very sol. H_2O. Spar. sol. AcOH. Insol. EtOH.
Methiodide: yellow needles from EtOH.Aq. M.p. 219–20° decomp.

Einhorn, *Ann.*, 1891, **265**, 221.
Einhorn, Liebrecht, *Ber.*, 1887, **20**, 1593.

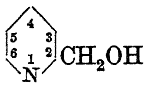
2-β-Pyridylacrylic Acid.
M.p. 233°.
B,HCl: m.p. 186–7°.
Et ester: b.p. 156–8°/14 mm. *Methiodide*: m.p. 147°.
Amide: m.p. 148°.

Panizzon, *Helv. Chim. Acta*, 1941, **24**, 24E.

2-γ-Pyridylacrylic Acid.
Reddish-brown cryst. from AcOEt. M.p. 296° decomp. Very sol. acids, alkalis. Spar. sol. H_2O. $KMnO_4$ ⟶ isonicotinic acid.
B,HCl: brown cryst. + $1H_2O$. M.p. 243–4°. Very sol. H_2O. Sol. EtOH. Spar. sol. Et_2O.
Acetate: cryst. from AcOH. M.p. 287–8°.
$B,HAuCl_4$: yellow needles. M.p. 235° decomp. Spar. sol. H_2O, EtOH. Insol. Et_2O.

Alberts, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1285.
Rabe, Kindler, *Ber.*, 1919, **52**, 1842.
Koelsch, *J. Am. Chem. Soc.*, 1943, **65**, 2460.
Kleiman, Weinhouse, *J. Org. Chem.*, 1945, **10**, 562.

Pyridylamine.
See Aminopyridine.
Pyridylbenzene.
See Phenylpyridine.
2-Pyridylcarbinol (*ω-Hydroxy-α-picoline, 2-hydroxymethylpyridine*)



C_6H_7ON MW, 109

Viscous oil with odour of pyridine. B.p. 112–13°/16 mm. Misc. with H_2O and most org. solvents.
B_2,H_2PtCl_6: reddish-yellow needles from EtOH. M.p. 179° decomp. Very sol. H_2O. Spar. sol. EtOH.
Picrate: yellow cryst. from EtOH. M.p. 159°. Spar. sol. cold EtOH.

Harries, Lénárt, *Ann.*, 1915, **410**, 107.

3-Pyridylcarbinol (*ω-Hydroxy-β-picoline, 3-hydroxymethylpyridine*).

B,HAuCl₄: yellow needles from H_2O. M.p. 136–7°.

B_2,H_2PtCl_6: brownish-red plates + $1H_2O$ from H_2O. M.p. 193–5°.

Picrate: yellow needles from H_2O. M.p. 158° (128°). Very sol. boiling H_2O, Me_2CO.

Benzoyl: b.p. 196–8°/17 mm. *Hydrochloride*: m.p. 116°.

Methochloride: m.p. 49°.

Methiodide: m.p. 159°.

Dehnel, *Ber.*, 1900, **33**, 3498.
Panizzon, *Helv. Chim. Acta*, 1941, **24**, 24E.

4-Pyridylcarbinol (*ω-Hydroxy-γ-picoline, 4-hydroxymethylpyridine*).

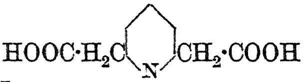
M.p. 47–50°. Very hygroscopic.

B,HCl: m.p. 175°.

Picrate: m.p. 137–9°.

Rabe, Spreckelsen, Wilhelm, Höter, *J. prakt. Chem.*, 1938, **151**, 65.

Pyridyl-2 : 6-diacetic Acid (*2 : 6-Di-[carboxymethyl]-pyridine*)



$C_9H_9O_4N$ MW, 195

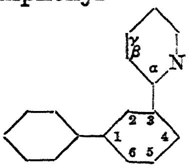
Cryst. + $1H_2O$. M.p. 140°.

Di-Me ester: $C_{11}H_{13}O_4N$. MW, 223. M.p. 64°.

Dianilide: m.p. 198°. *B_2,H_2PtCl_6*: M.p. 216°.

Oparina, *Chem. Zentr.*, 1935, I, 2536.

3-α-Pyridyldiphenyl



$C_{17}H_{13}N$ MW, 231

Viscous oil. B.p. 75–85°/0·002 mm. Absorption maximum: 2480 Å. in hexane.

Picrate: yellow prisms from Me_2CO. M.p. 169°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1281.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

3-β-Pyridyldiphenyl.

Viscous oil. B.p. 75–85°/0·002 mm. Absorption maximum: 2455 Å. in hexane.

Picrate: needles from Me_2CO. M.p. 178–9°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1281.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

3-γ-Pyridyldiphenyl.

Plates from pet. ether. M.p. 81–2°. Absorption maximum: 2480 Å. in hexane.

Picrate: needles from Me_2CO. M.p. 231°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

4-α-Pyridyldiphenyl.

Plates from EtOH. M.p. 141–2°. Absorption maximum: 2920 Å. in hexane.

Picrate: needles from Me_2CO. M.p. 186–7°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

4-β-Pyridyldiphenyl.

Plates from C_6H_6–pet. ether. M.p. 151–2°. Absorption maximum: 2790 Å. in hexane.

Picrate: needles from Me_2CO. M.p. 208–10°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

4-γ-Pyridyldiphenyl.

Plates from C_6H_6–pet. ether. M.p. 209°. Absorption maximum: 2790 Å. in hexane.

Picrate: needles from Me_2CO. M.p. 215°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1283.
Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

Pyridylethylene.
See Vinylpyridine.

Pyridyl Mercaptan.
See Mercaptopyridine.

Pyridylmethylamine.
See Aminopicoline.

Pyridyl-2-nitramine (*N-Nitro-2-aminopyridine, 2-nitraminopyridine*)

$C_5H_5O_2N_3$ MW, 139

Needles from H_2O. Decomp. at 184°. Sol. EtOH, hot H_2O, AcOEt. Sol. dil. alkalis. Spar. sol. strong min. acids. Insol. C_6H_6, ligroin.

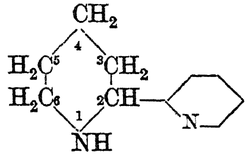
Tschitschibabin, Rasorenow, *J. Russ. Phys.-Chem. Soc.*, 1915, **47**, 1290.

Pyridyl-3-nitramine (*N-Nitro-3-aminopyridine, 3-nitraminopyridine*).

Small needles from H_2O. Decomp. at 170–5°. Sol. hot H_2O. Insol. C_6H_6. Aq. sol. reacts acid to litmus, neutral to Congo Red. Sol. dil. min. acids.

Tschitschibabin, Kirssanow, *Ber.*, 1927, **60**, 2435.

2-[2-Pyridyl]-piperidine (2-[2-*Piperidyl*]-*pyridine*)



$C_{10}H_{14}N_2$ MW, 162

B.p. 265–6°/756 mm. Sol. H_2O, org. solvents.
Picrate: prisms from H_2O. M.p. 187°.

Smith, *J. Am. Chem. Soc.*, 1931, **53**, 281.

3-[2-Pyridyl]-piperidine.
See Isoneonicotine.

2-[3-Pyridyl]-piperidine.
See Anabasine.

3-[3-Pyridyl]-piperidine.
See Nicotidine.

4-[3-Pyridyl]-piperidine (3-[4-*Piperidyl*]-*pyridine*).
Oil.
Picrate: long needles from H_2O. M.p. 240° slight decomp.

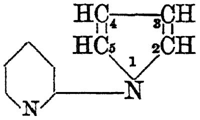
Smith, *J. Am. Chem. Soc.*, 1931, **53**, 282.

1-[4-Pyridyl]-piperidine (4-[1-*Piperidyl*]-*pyridine*).
Yellow cryst. M.p. 80°. B.p. 164°/13 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, dil. min. acids. Volatile in steam. Sublimes in vacuo at low temps.
B,HAuCl$_4$: orange plates. M.p. 161–3°.
Picrate: leaflets. M.p. 142°.
Methiodide: yellow cryst. from MeOH. M.p. 159°.

Graf, Lehmann, *J. prakt. Chem.*, 1933, **138**, 242.

4-[4-Pyridyl]-piperidine.
See Isonicotine.

1-[2-Pyridyl]-pyrrole



$C_9H_8N_2$ MW, 144

M.p. 17°. B.p. 250°/748 mm., 123°/11 mm. Gives intense blue col. with pine splinter moistened with HCl.
Chloroplatinate: yellow microcryst. powder + $2H_2O$. Slowly decomp. on heating.
Methiodide: m.p. 141–2°.
Picrate: m.p. 143°.

Tschitschibabin, Bylinkin, *Ber.*, 1923, **56**, 1745.
Wibaut, Dingemanse, *Chem. Abstracts*, 1923, **17**, 3873.

1-[4-Pyridyl]-pyrrole.
M.p. 80°.
Picrate: m.p. 224°.

Overhoff, *Rec. trav. chim.*, 1940, **59**, 741.

2-[2-Pyridyl]-pyrrole.
Cryst. from ligroin. M.p. 90° (87–8°). Intense blue col. with pine splinter moistened with HCl.
Methiodide: m.p. 188°.
Picrate: yellow cryst. M.p. 221° (143°).
N-*Me*: *see* 2 : 2′-Nicotyrine.

Tschitschibabin, Bylinkin, *Ber.*, 1923, **56**, 1745.
Wibaut, *Chem. Abstracts*, 1927, **21**, 3362.
Wibaut, *Rec. trav. chim.*, 1926, **45**, 657.

2-[3-Pyridyl]-pyrrole (*Nornicotyrine*).
Needles from ligroin-C_6H_6. M.p. 100–2°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin, H_2O. EtOH, Et_2O sols. show blue fluor. $FeCl_3 \longrightarrow$ orange-red col. Gives cryst. K salt.
B,HgCl$_2$: m.p. 178–9°.
B$_2$,*H*$_2$*PtCl*$_6$: yellow needles + $2H_2O$ from H_2O. Decomp. at 150°.
Methiodide: pale yellow needles from EtOH. M.p. 170–1°.
Picrate: yellow prisms from H_2O or EtOH. M.p. 202–3° (182°).
N-*Me*: *see* 3 : 2′-Nicotyrine.

Ehrenstein, *Arch. Pharm.*, 1931, **269**, 650.
Pictet, *Compt. rend.*, 1903, **137**, 861.
Pictet, Crépieux, *Ber.*, 1895, **28**, 1909.

2-[4-Pyridyl]-pyrrole.
M.p. 152°.
Picrate: m.p. 221°.

Overhoff, *Rec. trav. chim.*, 1940, **59**, 741.

3-[2-Pyridyl]-pyrrole.
M.p. 132°.
Methiodide: m.p. 146°.
Picrate: m.p. 193°.

Wibaut, *Chem. Abstracts*, 1927, **21**, 3362.
Wibaut, *Rec. trav. chim.*, 1926, **45**, 657.

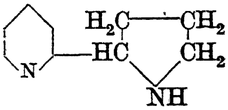
3-[3-Pyridyl]-pyrrole.
M.p. 140°.
Picrate: m.p. 198–9°.

Späth, Kainrath, *Ber.*, 1938, **71**, 1276.

3-[4-Pyridyl]-pyrrole.
M.p. 228°.
Picrate: m.p. 235°.

Overhoff, *Rec. trav. chim.*, 1940, **59**, 741.

2-[2-Pyridyl]-pyrrolidine (*α-Nornicotine*)



$C_9H_{12}N_2$ MW, 148

Oil. B.p. 120°/12 mm. Misc. with H_2O in all proportions. Sol. most org. solvents.
Picrate: cryst. from EtOH. M.p. 166°.
N-*Me*: α-nicotine. $C_{10}H_{14}N_2$. MW, 162. Oil. B.p. 122°/25 mm. Completely misc. with

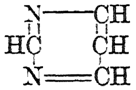
H_2O and most org. solvents. *Picrate*: cryst. from EtOH. M.p. 169°.

Craig, *J. Am. Chem. Soc.*, 1934, **56**, 1146.

2-[3-Pyridyl]-pyrrolidine.
See Nornicotine.

Pyridylsulphanilamide.
See Sulphapyridine.

Pyrimidine (1 : 3-*Diazine*)



$C_4H_4N_2$ MW, 80

Cryst. mass. M.p. 21°. B.p. 123·5–124°. Misc. with H_2O ⟶ neutral sol.

B,AuCl₃: needles. M.p. 226°.

Picrate: yellow needles. M.p. 156°.

Emery, *Ber.*, 1901, **34**, 4180.

Pyrimidone-imide.
See Aminopyrimidine.

Pyrimidylsulphanilamide.
See Sulphadiazine.

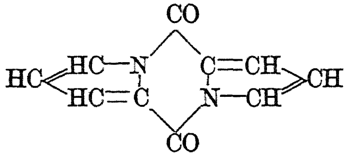
Pyrindole.
See Indolizine.

Pyrobromone.
See under Pyramidone.

Pyrocatechol.
See Catechol.

Pyrocinchonic Acid.
See Dimethylmaleic Acid.

Pyrocoll



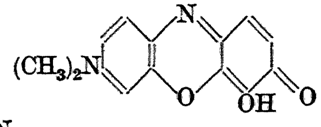
$C_{10}H_6O_2N_2$ MW, 186

From gelatine. Plates from AcOH. M.p. 268°. Sol. AcOEt, $CHCl_3$. Spar. sol. cold EtOH, Et_2O, C_6H_6, AcOH. Insol. H_2O. KOH ⟶ pyrrole-2-carboxylic acid.

Hale, Hoyt, *J. Am. Chem. Soc.*, 1916, **38**, 1065.
Ciamician, Silber, *Ber.*, 1884, **17**, 105.
Weidel, Ciamician, *Monatsh.*, 1880, **1**, 279.

Pyrogallic Acid.
See Pyrogallol.

Pyrogalline



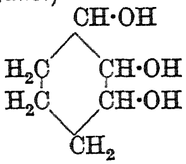
$C_{14}H_{12}O_3N_2$ MW, 256

Cryst. with greyish-green reflex from xylene. M.p. 240–1°. Sol. hot H_2O ⟶ bluish-violet sol. Sol. alkalis ⟶ reddish-violet sols. H_2SO_4 ⟶ red sol. HCl ⟶ blue sol. AcOH ⟶ blue sol.

Me ether: $C_{15}H_{14}O_3N_2$. MW, 270. Metallic green prisms from C_6H_6. M.p. 199–200°. Spar. sol. H_2O ⟶ blue sol. Gives cryst. salts with strong acids.

Kehrmann, Beyer, *Ber.*, 1912, **45**, 3341.

Pyrogallitol (1 : 2 : 3-*Trihydroxycyclohexane, hexahydropyrogallol*)



$C_6H_{12}O_3$ MW, 132

Three forms are known:

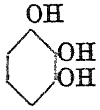
α-.
Needles from AcOEt or Me_2CO. M.p. 108°.
Tribenzoyl: needles from EtOH. M.p. 142°.

β-.
Cryst. from AcOEt. M.p. 124–5°.
Tribenzoyl: cryst. from AcOH. M.p. 184°.

γ-.
Cryst. from AcOH. M.p. 148°.
Tribenzoyl: cryst. from EtOH. M.p. 142°.

Lindemann, de Lange, *Ann.*, 1930, **483**, 31.

Pyrogallol (*Pyrogallic acid*, 1 : 2 : 3-*trihydroxybenzene*)



$C_6H_6O_3$ MW, 126

Plates and needles. M.p. 132·5–133·5°. B.p. 309°, 171·5°/12 mm. Sol. 2·25 parts H_2O at 13°. Sol. EtOH, Et_2O. Bitter taste. Heat of comb. 639 Cal. Rapidly absorbs O, turning brown. Reduces Pt, Au, Ag, Hg salts. Aq. sol. + Pd or enzymes ⟶ purpurogallin. Aq. sol. + $(NH_4)_2CO_3$ ⟶ pyrogallol-4-carboxylic acid and pyrogallol-4 : 6-dicarboxylic acid. Red. ⟶ 1 : 2 : 3-cyclohexantriol. CH_3COCl ⟶ triacetyl deriv. Alk. sol. + $FeCl_3$ ⟶ deep red col. H·CHO + HCl ⟶ bright red col., ppt. on boiling. Used as developer in photography, in medicine, and for gas analysis. Is a blood poison.

Monoacetyl: viscous oil. B.p. 185°/25 mm.
Diacetyl: m.p. 110–11°. Sol. alkalis.
Triacetyl: m.p. 165°.
Monobenzoyl: prisms from AcOH or $CHCl_3$. M.p. 140°. Very sol. EtOH, Et_2O. Insol. H_2O. $FeCl_3$ ⟶ green col.
Dibenzoyl: needles from toluene. M.p. 108°. Very sol. EtOH, Et_2O. Insol. H_2O. No col. with $FeCl_3$.
Tribenzoyl: prisms from EtOH. M.p. 89–90°.

Tri-phenylurethane: needles from EtOH–Et_2O. M.p. 173°. Very sol. EtOH. Spar. sol. Et_2O, $CHCl_3$, C_6H_6.

Tri-diphenylurethane: m.p. 211·5–212·5°.

Tricinnamoyl: cryst. from EtOH. M.p. 136°.

$C_6H_6O_3,C_6H_3(NO_2)_3$-1 : 3 : 5: yellow needles. M.p. 163°.

1-*Me ether*: $C_7H_8O_3$. MW, 140. Needles. M.p. 38–41°. B.p. 129°/10 mm. *Diacetyl*: plates from EtOH. M.p. 91–3°. Spar. sol. EtOH. *Dibenzoyl*: cryst. from EtOH. M.p. 156–8°.

2-*Me ether*: cryst. from C_6H_6. M.p. 85–7°. B.p. 154–5°/24 mm. *Diacetyl*: plates from EtOH. M.p. 51–4°.

1 : 2-*Di-Me ether*: $C_8H_{10}O_3$. MW, 154. Clear oil. B.p. 233–4°, 124–5°/17 mm. *Benzoyl*: needles from pet. ether. M.p. 55–7°.

1 : 3-*Di-Me ether*: prisms from H_2O. M.p. 55–6°. B.p. 262–7°. Sol. 57 parts H_2O at 13°. *Acetyl*: cryst. from EtOH.Aq. M.p. 53·5°. *Benzoyl*: prisms. M.p. 118°.

Tri-Me ether: $C_9H_{12}O_3$. MW, 168. Needles from EtOH.Aq. M.p. 47°. B.p. 241°. D_{45}^{45} 1·1118. Very sol. EtOH, Et_2O, C_6H_6. $C_9H_{12}O_3,C_6H_3(NO_2)_3$-1 : 3 : 5: yellow prisms. M.p. 81°.

Mono-Et ether: $C_8H_{10}O_3$. MW, 154. Needles. M.p. 95° (102–4°). Very sol. EtOH, Et_2O. Spar. sol. cold H_2O, C_6H_6.

1 : 3-*Di-Et ether*: $C_{10}H_{14}O_3$. MW, 182. Needles. M.p. 79–80°. B.p. 263–5°. Very sol. C_6H_6. Spar. sol. cold EtOH.Aq.

Tri-Et ether: $C_{12}H_{18}O_3$. MW, 210. Cryst. from EtOH.Aq. M.p. 39°. Insol. alkalis.

Triallyl ether: m.p. 3·5–4°. D_4^{20} 1·04. n_D^{20} 1·5265.

Marks, B.P. 140,694, (*Chem. Abstracts*, 1920, **14**, 2203).
Ullmann, *Ann.*, 1903, **327**, 116.
Einhorn, Hollandt, *Ann.*, 1898, **301**, 106.
Luynes, Esperandieu, *Compt. rend.*, 1865, **61**, 487.

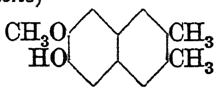
Pyrogallol-dicarboxylic Acid.

See 4 : 5 : 6-Trihydroxyisophthalic Acid *and* 3 : 4 : 5-Trihydroxyphthalic Acid.

Pyroglutamic Acid.

See Glutiminic Acid.

Pyroguaiacin (6-*Hydroxy*-7-*methoxy*-2 : 3-*dimethylnaphthalene*)



$C_{13}H_{14}O_2$ MW, 202

Plates from EtOH. M.p. 183°. Mod. sol. Et_2O. Spar. sol. boiling H_2O, EtOH. Sublimes. Zn dust. dist. ⟶ 2 : 3-dimethylnaphthalene. H_2SO_4 ⟶ dark blue col. Gives cryst. Na and K salts.

Acetyl: needles from EtOH. M.p. 122–4°.

Benzoyl: cryst. from AcOH. M.p. 179°.

6-*Me ether*: 6 : 7-dimethoxy-2 : 3-dimethylnaphthalene. $C_{14}H_{16}O_2$. MW, 216. Plates from EtOH or formic acid, needles from ligroin. M.p. 149–50°. Spar. sol. EtOH, formic acid. Ox. ⟶ 6 : 7-dimethoxy-2 : 3-dimethyl-1 : 4-naphthoquinone. *Picrate*: red needles from MeOH. M.p. 130–1°.

Haworth, Mavin, *J. Chem. Soc.*, 1932, 1488.
Schroeter, Lichtenstadt, Irineu, *Ber.*, 1918, **51**, 1604.

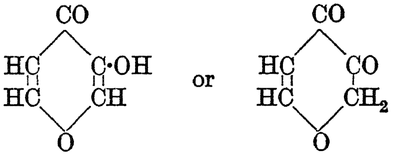
Pyrohypaconitine.

See under Hypaconitine.

Pyroiodone.

See under Pyramidone.

Pyromeconic Acid (3-*Hydroxy*-γ-*pyrone*)



$C_5H_4O_3$ MW, 112

Colourless prisms from H_2O or EtOH. M.p. 117°. B.p. 227–8°. Very sol. H_2O, EtOH. Sol. $CHCl_3$. Spar. sol. Et_2O. Possesses slight acid properties. Sublimes. $FeCl_3$ ⟶ blood-red col. Forms unstable salts decomp. by H_2O. Does not combine with hydroxylamine. Gives cryst., spar. sol. ferric salt. Gives unstable hydrochloride. Red. ⟶ 3 : 4-dihydroxytetrahydropyran.

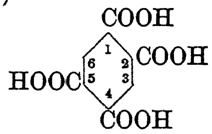
Acetyl: prisms from EtOH. M.p. 91°. Very sol. H_2O, EtOH, $CHCl_3$. No col. with $FeCl_3$.

Me ether: $C_6H_6O_2$. MW, 126. Cryst. from C_6H_6–ligroin. M.p. 85°. Sol. boiling ligroin. Very sol. cold H_2O, hot C_6H_6, Et_2O. Sublimes in vacuo.

Et ether: $C_7H_8O_3$. MW, 140. Colourless oil. B.p. 220–1°.

Borsche, *Ber.*, 1916, **49**, 2544.
Ost, *J. prakt. Chem.*, 1879, **19**, 183.
Garkusha, *J. Gen. Chem. U.S.S.R.*, 1946, **16**, 2025, (*Chem. Abstracts*, 1948, **42**, 566).

Pyromellitic Acid (*Benzene*-1 : 2 : 4 : 5-*tetracarboxylic acid*)



$C_{10}H_6O_8$ MW, 254

Prisms + $2H_2O$ from H_2O. M.p. anhyd. 275°.

1 : 4-*Di-Me ester*: $C_{12}H_{10}O_8$. MW, 282. M.p. 176–7°.

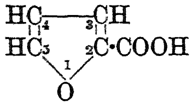
Tetra-Me ester: $C_{14}H_{14}O_8$. MW, 310. Leaflets from MeOH. M.p. 141·5°.

Tetra-Et ester: $C_{18}H_{22}O_8$. MW, 366. M.p. 54°.

Di-anhydride: $C_{10}H_2O_6$. MW, 218. Needles. M.p. 286°. Sol. Me_2CO, AcOEt. Insol. Et_2O, $CHCl_3$, ligroin. Sublimes.

Philippi, Thelen, *Organic Syntheses*, 1930, X, 90.
v. Braun, Lemke, *Ber.*, 1924, **57**, 682.
de Diesbach, Guhl, *Helv. Chim. Acta*, 1927, **10**, 448.
Schroeter, *Ber.*, 1924, **57**, 2023.
Fieser, Herschberg, *J. Am. Chem. Soc.*, 1935, **57**, 1508.
I.G., D.R.P., 563,129, (*Chem. Abstracts*, 1933, **27**, 1008).
Feist, *Ber.*, 1911, **44**, 137.
Silberrad, *J. Chem. Soc.*, 1906, **89**, 1795.
Clar, *Ber.*, 1942, **75**, 1330.

Pyromucic Acid (*Furan-2-carboxylic acid, α-furoic acid*)



$C_5H_4O_3$ MW, 112

Leaflets from H_2O. M.p. 133–4°. B.p. 230–2°, 141–4°/20 mm. Part. sublimes at 100°, easily at 130–40°/50–60 mm. Sol. to 2·7% in H_2O at 0°. Sol. 28 parts H_2O at 15°, 4 parts at boil. Sol. EtOH, Et_2O. $k = 7{\cdot}6\ (7{\cdot}07) \times 10^{-4}$ at 25°. Heat of comb. C_v 494·4 Cal. At 275° under press. quantitatively ⟶ furan + CO_2. MnO_2 + conc. HCl ⟶ mucochloric acid.

Me ester: $C_6H_6O_3$. MW, 126. B.p. 181·3°. $D_4^{21\cdot4}$ 1·1786. n_D^{20} 1·4860.

Et ester: $C_7H_8O_3$. MW, 140. Leaflets. M.p. 34°. B.p. 195°/766 mm., 128°/95 mm. $D_4^{20\cdot8}$ 1·1174, D_4^{40} 1·0974. $n_D^{20\cdot8}$ 1·4797.

Propyl ester: $C_8H_{10}O_3$. MW, 154. B.p. 210·9°. $D_4^{25\cdot9}$ 1·0745. $n_D^{25\cdot9}$ 1·4737.

Isopropyl ester: b.p. 198·6°. $D_4^{23\cdot7}$ 1·0655. $n_D^{23\cdot7}$ 1·4682.

Butyl ester: $C_9H_{12}O_3$. MW, 168. B.p. 83–4°/1 mm. D_4^{20} 1·0555.

Isobutyl ester: b.p. 220·8–222·6°. $D_4^{27\cdot5}$ 1·0383. $n_D^{27\cdot5}$ 1·4676.

n-*Amyl ester*: $C_{10}H_{14}O_3$. MW, 182. B.p. 95–7°/1 mm. D_4^{20} 1·0335.

n-*Hexyl ester*: $C_{11}H_{16}O_3$. MW, 196. B.p. 105–7°/1 mm. D_4^{20} 1·0170.

n-*Heptyl ester*: $C_{12}H_{18}O_3$. MW, 210. B.p. 116–17°/1 mm. D_4^{20} 1·0005.

Cyclohexyl ester: m.p. 32–3°. B.p. 122–4°. n_D^{30} 1·4499.

Allyl ester: b.p. 206–9°.

Phenyl ester: $C_{11}H_8O_3$. MW, 188. Prisms from EtOH. M.p. 42°.

Benzyl ester: b.p. 141–2°/12 mm. n_D^{30} 1·5505.

p-*Nitrobenzyl ester*: cryst. from 70% EtOH. M.p. 133·5°.

Anhydride: $C_{10}H_6O_5$. MW, 206. Needles from EtOH. M.p. 73°. B.p. 325° part. decomp.

Chloride: $C_5H_3O_2Cl$. MW, 130·5. M.p. − 2°. B.p. 173°, 66°/10 mm. Stable to H_2O.

Amide: $C_5H_5O_2N$. MW, 111. M.p. 142–3°. Part. sublimes at 100°.

Methylamide: $C_6H_7O_2N$. MW, 125. Cryst. from ligroin. M.p. 64°. B.p. 250–3°.

Ethylamide: $C_7H_9O_2N$. MW, 139. B.p. 258°.

Nitrile: C_5H_3ON. MW, 93. B.p. 146°/738 mm. D_4^{20} 1·0822. n_D^{20} 1·4798.

Anilide: prisms from C_6H_6. M.p. 123·5°.

o-*Toluidide*: prisms from EtOH. M.p. 62°.

m-*Toluidide*: prisms from EtOH. M.p. 87°.

p-*Toluidide*: prisms from EtOH. M.p. 107·5°.

Wilson, *Organic Syntheses*, Collective Vol. I, 270.
Gennari, *Gazz. chim. ital.*, 1894, **24**, ii, 249.
Hughes, Johnson, *J. Am. Chem. Soc.*, 1931, **53**, 744.
Trickey, Miner, U.S.P., 1,665,236, (*Chem. Abstracts*, 1928, **22**, 1783).
Zanetti, Beckmann, *J. Am. Chem. Soc.*, 1926, **48**, 1068.

Pyromucylacetic Acid.

See α-Furoylacetic Acid.

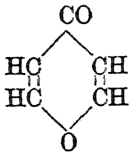
Pyromucylformic Acid.

See α-Furoylformic Acid.

α-Pyrone.

See Coumalin.

γ-Pyrone



$C_5H_4O_2$ MW, 96

Hygroscopic cryst. M.p. 32·5°. B.p. 215°, 105°/23 mm., 97°/13 mm. Very sol. H_2O, Et_2O, $CHCl_3$, AcOH. Sol. C_6H_6. Spar. sol. pet. ether, CS_2. Sol. KOH.Aq. with yellow col. Non-volatile in steam. NH_3 at 120–40° ⟶ 4-hydroxypyridine.

$C_5H_4O_2,HCl$: prisms from EtOH. M.p. 139°.

$C_5H_4O_2,(COOH)_2$: plates. M.p. 136·5°.

$3C_5H_4O_2,(COOH)_2$: prisms. M.p. 139°.

$3C_5H_4O_2,HAuCl_4$: leaflets. M.p. 116·5°.

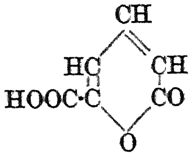
Picrate: needles from H_2O or EtOH. M.p. 129°.

Willstätter, Pummerer, *Ber.*, 1904, **37**, 3745; 1905, **38**, 1465.
Ost, *J. prakt. Chem.*, 1884, **29**, 63.

α-Pyrone-5-carboxylic Acid.

See Coumalic Acid.

α-Pyrone-6-carboxylic Acid



$C_6H_4O_4$ MW, 140

Prisms from H_2O. M.p. 228°. Sublimes in needles. Spar. sol. EtOH, Me_2CO, hot H_2O. Insol. $CHCl_3$, C_6H_6, pet. ether. Reduces $NH_3.AgNO_3$.

Et ester: $C_8H_8O_4$. MW, 168. Plates from ligroin. M.p. 59–60°. Sol. H_2O and most org. solvents.

Lapworth, *J. Chem. Soc.*, 1901, **79**, 1280.
Gault, *Compt. rend.*, 1914, **159**, 73.

γ-Pyrone-2-carboxylic Acid.
See Comanic Acid.

γ-Pyrone-2 : 6-dicarboxylic Acid.
See Chelidonic Acid.

Pyroracemic Acid.
See Pyruvic Acid.

Pyroracemic Alcohol.
See Hydroxyacetone.

Pyroracemic Aldehyde.
See Pyruvic Aldehyde.

Pyrotartaric Acid.
See Methylsuccinic Acid.

Pyroterebic Acid (*3-Methyl-2-butylene-1-carboxylic acid, 2-isopropylidene-propionic acid, 2-isohexenic acid*)

$(CH_3)_2C{:}CH{\cdot}CH_2{\cdot}COOH$

$C_6H_{10}O_2$ MW, 114

Oil with somewhat sharp odour. B.p. 207–8°, 111°/22 mm. Readily converted to isocaprolactone. $k = 2{\cdot}51 \times 10^{-5}$ at 25°.

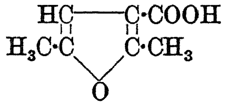
Et ester: $C_8H_{14}O_2$. MW, 142. B.p. 58°/11 mm. D_4^{17} 0·9134. n_D^{17} 1·4329.

Nitrile: C_6H_9N. MW, 95. B.p. 166°, 65°/20 mm. Fuming HCl ⟶ isocaprolactone.

Anilide: needles from C_6H_6. M.p. 106°.

Fittig, Geisler, *Ann.*, 1881, **208**, 39.
Linstead, *J. Chem. Soc.*, 1929, 2506.

Pyrotritaric Acid (*Uvinic acid, 2 : 5-dimethyl-furan-3-carboxylic acid, 2 : 5-dimethyl-β-furoic acid*)



$C_7H_8O_3$ MW, 140

Needles from hot H_2O. M.p. 135°. Sol. EtOH, Et_2O. Mod. sol. hot H_2O. Sublimes. Volatile in steam. Chromic mixture ⟶ CO_2 + $CH_3{\cdot}COOH$. Dil. HNO_3 ⟶ CO_2 + oxalic acid.

Me ester: $C_8H_{10}O_3$. MW, 154. B.p. 198°.

Et ester: $C_9H_{12}O_3$. MW, 168. Oil. B.p. 208–9°, 99–101°/14 mm., 83–5°/6 mm. $D_4^{23\cdot1}$ 1·0478. $n_D^{23\cdot1}$ 1·4686.

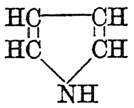
Hydrazide: cryst. from H_2O. M.p. 136°.

Gilman, Burtner, *Rec. trav. chim.*, 1932, **51**, 667.
Scott, Johnson, *J. Am. Chem. Soc.*, 1932, **54**, 2555.

Pyrrocoline.
See Indolizine.

Pyrrodiazole.
See 1 : 2 : 4-Triazole.

Pyrrole



C_4H_5N MW, 67

Present in coal tar. Colourless liq. with characteristic odour. B.p. 130–1°/761 mm. D_4^{20} 0·9691. n_D^{20} 1·5085. Heat of comb. C_v 567·5 Cal. Sol. EtOH, Et_2O. Spar. sol. H_2O. Turns brown in air. Forms metallic salts. CrO_3 + H_2SO_4 ⟶ maleinimide. Polymerises with HCl to a trimer. Nitroprussiates in alkali ⟶ red col. which turns blue on addn. of acid.

N-*Me*: *see N*-Methylpyrrole.

N-*Et*: C_6H_9N. MW, 95. Liq. with odour resembling pyrrole. B.p. 131°. D^{16} 0·8881. Misc. with EtOH, Et_2O.

N-*Propyl*: $C_7H_{11}N$. MW, 109. B.p. 146·5–147·5°. D_4^{20} 0·8833.

N-*Butyl*: $C_8H_{13}N$. MW, 123. B.p. 165–80°.

N-*Isoamyl*: $C_9H_{15}N$. MW, 137. B.p. 180–4°. D^{10} 0·8786.

N-*Allyl*: C_7H_9N. MW, 107. B.p. 105°/48 mm. Unstable in air. Volatile in steam.

N-*Phenyl*: *see* 1-Phenylpyrrole.

N-o-*Tolyl*: oil. B.p. 246°. Spar. sol. boiling H_2O. Volatile in steam.

N-p-*Tolyl*: leaflets from EtOH. M.p. 82°. B.p. 252°/728·5 mm. Very sol. EtOH, $CHCl_3$, C_6H_6, pet. ether. Spar. sol. boiling H_2O. Volatile in steam.

N-*Benzyl*: b.p. 245–6°, 138–9°/27 mm. Sol. EtOH, Et_2O. Insol. H_2O. Turns yellow in light and air.

N-1-*Naphthyl*: needles from EtOH.Aq. M.p. 42°. B.p. above 360°. Volatile in steam.

N-2-*Naphthyl*: cryst. from EtOH.Aq. M.p. 107°. B.p. above 360°. Volatile in steam.

N-*Formyl*: b.p. 39°/22 mm.

N-*Acetyl*: b.p. 181–2°. Insol. H_2O.

N-*Propionyl*: yellowish oil. B.p. 192–4°.

N-*Benzoyl*: yellowish oil. B.p. 276°/715 mm. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6, pet. ether. Insol. H_2O. Volatile in steam.

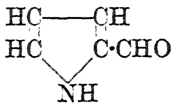
Phenylurethane: m.p. 142–3°.

α-Naphthylurethane: m.p. 162–162·5°.

Picrate: orange-red cryst. M.p. 69° decomp.

McElvain, Bolliger, *Organic Syntheses*, Collective Vol. I, 461.
Pictet, *Ber.*, 1904, **37**, 2792.
Jurjew, *Ber.*, 1936, **69**, 440.
Jurjew, Schenjan, *Chem. Zentr.*, 1936, I, 4293.
Fischer, Orth, *Die Chemie des Pyrrols*, Vol. I (1934); Vol. II, Part 1 (1937); Vol. II, Part 2 (1940).

Pyrrole-2-aldehyde (*2-Formylpyrrole*)



C_5H_5ON MW, 95

Prisms from pet. ether. M.p. 46–7°. B.p. 217–19°, 114°/15 mm. n_D^{16} 1·5939. Heat of comb. C_p 616·7 Cal. Volatile in steam. Does not oxidise in air.

Oxime: needles from $CHCl_3$ or C_6H_6. M.p. 164·5°.

Phenylhydrazone: needles from ligroin. M.p. 139–139·5°.

p-*Nitrophenylhydrazone*: red needles from xylene. M.p. 182·5–183°.

Semicarbazone: leaflets from H_2O. M.p. 183·5°.

N-*Me*: *see N*-Methylpyrrole-2-aldehyde.

N-*Butyl*: $C_9H_{13}ON$. MW, 151. B.p. about 75°/2 mm. *Semicarbazone*: cryst. from EtOH.Aq. M.p. 146–147·5°.

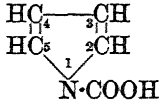
N-*Isoamyl*: $C_{10}H_{15}ON$. MW, 165. B.p. about 90°/2 mm. *Semicarbazone*: cryst. from EtOH.Aq. M.p. 155–7°.

N-*Phenyl*: m.p. 120°. Decomp. in sunlight.

N-*Benzoyl*: cryst. from ligroin. M.p. 90°. *Phenylhydrazone*: greenish-yellow plates from EtOH. M.p. 154°.

Emmert, Diehl, Gollwitzer, *Ber.*, 1929, **62**, 1737.
Putochin, *Ber.*, 1926, **59**, 1992.
Emmert, Diehl, *Ber.*, 1931, **64**, 131.
Reichstein, *Helv. Chim. Acta*, 1930, **13**, 352.
Alessandri, Passerini, *Gazz. chim. ital.*, 1921, **51**, 262.

Pyrrole-N-carboxylic Acid (*Pyrrole-1-carboxylic acid*)



$C_5H_5O_2N$ MW, 111

Prisms from Et_2O. M.p. 95° decomp. Sol. EtOH, Et_2O. Mod. sol. $CHCl_3$. Spar. sol. H_2O, C_6H_6, ligroin. At m.p. decomp. quantitatively into pyrrole + CO_2. Resinifies with min. acids.

Et ester: $C_7H_9O_2N$. MW, 139. Liq. with characteristic odour. B.p. 180°/770 mm. Insol. H_2O.

Amide: $C_5H_6ON_2$. MW, 110. Leaflets from H_2O. M.p. 166°. Sublimes.

Tschelinzeff, Maxoroff, *Ber.*, 1927, **60**, 196.
Ciamician, Magnaghi, *Ber.*, 1885, **18**, 416.

Pyrrole-2-carboxylic Acid (*Pyrrole-α-carboxylic acid*).

Leaflets from H_2O. M.p. 208·5 (192°) decomp. ⟶ pyrrole + CO_2. Sol. H_2O, EtOH, Et_2O. k (acid) = $4{\cdot}0 \times 10^{-5}$ at 25°: k (base) = 3×10^{-13}. Isoelectric point 2·9. The esters possess marked local anæsthetic properties.

Me ester: $C_6H_7O_2N$. MW, 125. Needles from pet. ether. M.p. 73°. B.p. 220–3°/740 mm., 120–30°/20–30 mm., 115–20°/12–16 mm. Sol. EtOH, Et_2O. Spar. sol. H_2O, pet ether.

Et ester: $C_7H_9O_2N$. MW, 139. Cryst. M.p. 39°. B.p. 230–2°. Very sol. EtOH, Et_2O, C_6H_6, pet. ether. Very spar. sol. H_2O.

Propyl ester: $C_8H_{11}O_2N$. MW, 153. B.p. 164–7°/50 mm.

Butyl ester: $C_9H_{13}O_2N$. MW, 167. M.p. 36–8°. B.p. 255–60°/740 mm.

Isobutyl ester: m.p. 68–9°. B.p. 250–5°/740 mm., 119–22°/70 mm.

Isoamyl ester: $C_{10}H_{15}O_2N$. MW, 181. B.p. 186–90°/100 mm.

Chloride: α-pyrroyl chloride. C_5H_4ONCl. MW, 129·5. Cryst. from Et_2O–ligroin. Sinters at 110°, decomp. at higher temps. Very sol. Et_2O, $CHCl_3$. Decomp. rapidly in moist air.

Amide: $C_5H_6ON_2$. MW, 110. Plates from H_2O or EtOH. M.p. 176°. Sweet taste.

Anilide: prisms from EtOH.Aq. M.p 153–4°.

N-*Me*: $C_6H_7O_2N$. MW, 125. M.p. 135°. *Methylamide*: prisms. M.p. 89–90°. Volatile in steam.

N-*Et*: $C_7H_9O_2N$. MW, 139. Needles from H_2O. M.p. 78°. *Ethylamide*: prisms from H_2O. M.p. 43–4°. B.p. 269–70°.

N-*Phenyl*: *see* 1-Phenylpyrrole-2-carboxylic Acid.

Hydrazide: cryst. from EtOH.Aq. M.p. 231–2° decomp.

Azide: cryst. M.p. 105° decomp.

Oddo, *Chem. Abstracts*, 1925, **19**, 2492.
Blicke, Blake, *J. Am. Chem. Soc.*, 1930, **52**, 238.
Schwanert, *Ann.*, 1860, **116**, 272.
Ciamician, Silber, *Ber.*, 1884, **17**, 104, 1152.
Bell, *Ber.*, 1877, **10**, 1866.
Fischer, van Slyke, *Ber.*, 1911, **44**, 3169.
Oddo, Moschini, *Gazz. chim. ital.*, 1912, **42**, ii, 254.

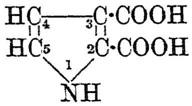
Pyrrole-3-carboxylic Acid (*Pyrrole-β-carboxylic acid*).

Needles. M.p. 161–2° decomp. Heat in vacuo ⟶ pyrrole + CO_2.

Me ester: needles or leaflets. M.p. 129°. Sol. EtOH, Et_2O. Spar. sol. pet. ether.

Dennstedt, Zimmermann, *Ber.*, 1887, **20**, 855.
Ciamician, Silber, *Ber.*, 1884, **17**, 1438.
Oddo, Moschini, *Gazz. chim. ital.*, 1912, **42**, ii, 255.

Pyrrole-2 : 3-dicarboxylic Acid



$C_6H_5O_4N$ MW, 155

3-*Me ester*: needles from EtOH. M.p. 200°.

Rinkes, *Rec. trav. chim.*, 1938, **57**, 423.

Pyrrole-2 : 5-dicarboxylic Acid.
Needles from EtOH.Aq. Decomp. about 260°, part. ⟶ pyrrole + CO_2. Sol. Et_2O, Me_2CO. Insol. $CHCl_3$, AcOEt, C_6H_6, pet. ether.

Me ester: $C_7H_7O_4N$. MW, 169. M.p. 243°.

Di-Me ester: $C_8H_9O_4N$. MW, 183. Needles from H_2O. M.p. 132°. Sol. EtOH, Et_2O, C_6H_6.

Di-Et ester: $C_{10}H_{13}O_4N$. MW, 211. Needles. M.p. 82°.

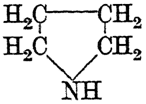
N-*Me*: $C_7H_7O_4N$. MW, 169. Cryst. from H_2O. Sinters and sublimes with part. decomp. about 275°. *Di-Me ester*: $C_9H_{11}O_4N$. MW, 197. Needles from EtOH.Aq. M.p. 80–1°.

N-*Et*: $C_8H_9O_4N$. MW, 183. Needles from EtOH.Aq. Decomp. at 250° without melting ⟶ *N*-ethylpyrrole + CO_2. Insol. H_2O. *Diethylamide*: needles. M.p. 229–30°.

N-*Phenyl*: $C_{12}H_9O_4N$. MW, 231. Cryst. from H_2O. M.p. 235–40° ⟶ *N*-phenylpyrrole + CO_2. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Insol. pet. ether. *Mono-Me ester*: m.p. 110°.

Ciamician, Silber, *Ber.*, 1887, **20**, 2595; 1886, **19**, 1960.
Tschelinzew, Maxorow, *Chem. Zentr.*, 1923, I, 1507.
Bell, *Ber.*, 1877, **10**, 1864.

Pyrrolidine (*Tetrahydropyrrole, tetramethyleneimine*)



C_4H_9N MW, 71

Present in tobacco leaves. Liq. with odour resembling piperidine. B.p. 88·5–89°. $D^{22.5}$ 0·8520. n_D^{15} 1·4270. Fumes in air. Misc. with H_2O. Strongly alkaline.

B,HAuCl₄: yellow cryst. from H_2O. M.p. 206° decomp.

B_2,2HI,CdI$_2$: leaflets or needles from H_2O. M.p. 217–19° (200–2°).

B_2,H_2PtCl_6: orange prisms from EtOH.Aq. M.p. about 200° decomp.

Picrate: (i) *B*,$C_6H_3O_7N_3$: yellow needles from EtOH. M.p. 112°. (ii) *B_2*,$C_6H_3O_7N_3$: dark red cryst. M.p. 163–4°.

N-*Me*: *see* *N*-Methylpyrrolidine.

N-*Et*: $C_6H_{13}N$. MW, 99. B.p. 104°. D_4^{20} 0·8156. n_D^{15} 1·4113. *Picrate*: leaflets. M.p. 186°.

N-*Propyl*: $C_7H_{15}N$. MW, 113. B.p. 130°. D_4^{20} 0·8171. n_D^{20} 1·4389. Sol. H_2O. *Picrate*: m.p. 105° (101°).

N-*Butyl*: $C_8H_{17}N$. MW, 127. Slightly sol. H_2O. *Chloroaurate*: yellow leaflets from H_2O. M.p. 78°. *Picrate*: m.p. 124°.

N-*Amyl*: $C_9H_{19}N$. MW, 141. B.p. 179°, 89–94°/28 mm. D_4^{20} 0·8216. n_D 1·44276. *Chloroplatinate*: prisms from EtOH. M.p. 145°. *Picrate*: reddish-yellow needles from EtOH. M.p. 118–19°. *Methiodide*: m.p. 169–70°.

N-*Isoamyl*: b.p. 78°/32 mm. D_4^{20} 0·8137. n_D 1·43994. *Chloroaurate*: m.p. 158°.

N-*Allyl*: $C_7H_{13}N$. MW, 111. Volatile in steam and ether vapour. *Chloroaurate*: m.p. 97–8°. *Chloroplatinate*: m.p. 205°.

N-*Cyclohexyl*: $C_{10}H_{19}N$. MW, 153. B.p. 79°/8 mm. D^{20} 0·9315. n_D^{20} 1·4848.

N-*Benzyl*: $C_{11}H_{15}N$. MW, 161. B.p. 237°. Sol. EtOH, Et_2O. Absorbs CO_2 and H_2O from the air. *B,HAuCl₄*: yellow prisms. M.p. 120° decomp. *B_2,H_2PtCl_6*: yellowish-red needles. M.p. 156° decomp. *Picrate*: m.p. 128°.

N-*Phenyl*: *see* 1-Phenylpyrrolidine.

N-p-*Nitrophenyl*: m.p. 167–8°.

N-o-*Tolyl*: $C_{11}H_{15}N$. MW, 161. B.p. 103–4°/9 mm. D_4^{20} 0·9981. n_D^{20} 1·5658.

N-p-*Tolyl*: m.p. 41·5°. B.p. 126–7°/9 mm. D_4^{50} 0·9728. n_D^{50} 1·5583. *Picrate*: m.p. 111°.

N-*Benzoyl*: viscous liq. B.p. 190–1°/12 mm. Insol. H_2O and dil. acids. Dist. with PCl_5 ⟶ benzonitrile + 1 : 4-dichlorobutane.

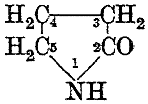
N-*Nitroso*: yellow oil. B.p. 214° decomp.

N-p-*Toluenesulphonyl*: cryst. from EtOH. M.p. 123°.

Schlinck, *Ber.*, 1899, **32**, 951.
v. Braun, Beschke, *Ber.*, 1906, **39**, 4121.
Ladenburg, *Ber.*, 1887, **20**, 442.
v. Braun, *Ber.*, 1911, **44**, 1252; 1916, **49**, 2642.
de Jong, Wibaut, *Rec. trav. chim.*, 1930, **49**, 237.
Yur'ev, Shen'yan, *Chem. Abstracts*, 1935, **29**, 3335.
Ochiai, Tsuda, *Ber.*, 1934, **67**, 1017.

Pyrrolidine-2-carboxylic Acid.
See Proline.

2-Pyrrolidone (*2-Ketopyrrolidine, butyrolactam, anhydride or lactam of piperidinic acid*)



C_4H_7ON MW, 85

Cryst. from pet. ether. M.p. 24·6°. B.p. 245°, 133°/12 mm. (114°/14 mm.). D_4^{20} 1·120, D_4^{40} 1·097. Spar. volatile in steam. Aq. sol. reacts

neutral to litmus. Very sol. H_2O, EtOH, Et_2O, $CHCl_3$, AcOEt, CS_2, C_6H_6. Spar. sol. pet. ether. In moist air ⟶ a monohydrate.

B,H_2O: plates. M.p. 35° (29·7–29·9°).

B_2,HCl: cryst. from Me_2CO. M.p. 86–8°.

B,HCl: cryst. from Me_2CO. M.p. 128–31°.

B_2,HBr: cryst. from $CHCl_3$ or Me_2CO. M.p. 135–7°.

B,HBr: cryst. M.p. 108–21°.

B_2,$HAuCl_4$: yellow cryst. from H_2O. M.p. 82°.

N-*Me*: *see* *N*-Methyl-2-pyrrolidone.

N-*Et*: $C_6H_{11}ON$. MW, 113. Oil. B.p. 218°/751 mm.

N-*Isopropyl*: $C_7H_{13}ON$. MW, 127. Oil with peppermint odour. B.p. 221–2°/736 mm. Volatile.

N-*Butyl*: $C_8H_{15}ON$. MW, 141. B.p. 121°/16 mm. D_4^{20} 0·964. n_D^{20} 1·4650.

N-*Amyl*: $C_9H_{17}ON$. MW, 155. B.p. 87–88·5°/1 mm. n_D^{25} 1·4619.

N-*Phenyl*: *see* 1-Phenylpyrrolidone-2.

N-p-*Tolyl*: needles from H_2O. M.p. 88·5°. B.p. 189°/13 mm.

N-*Acetyl*: b.p. 231°/737 mm.

Tafel, Stern, *Ber.*, 1900, **33**, 2226.
Gabriel, *Ber.*, 1889, **22**, 3338.
Späth, Breusch, *Monatsh.*, 1928, **50**, 356.

3-Pyrrolidone (3-*Ketopyrrolidine*)

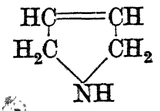
N-*Me*: *see* *N*-Methyl-3-pyrrolidone.

Cook, Reed, *J. Chem. Soc.*, 1945, 399.

2-Pyrrolidone-5-carboxylic Acid.

See Glutiminic Acid.

3-Pyrroline (*Dihydropyrrole*)



C_4H_7N MW, 69

Liq. Fumes in air. B.p. 90–1°/750·5 mm. D_4^{20} 0·9097. n_D^{20} 1·4664. Sol. H_2O. Absorbs CO_2 and H_2O from the air.

B,HCl: prisms from EtOH. M.p. 173–4°.

B,$HAuCl_4$: yellow prisms from H_2O. M.p. 152°.

B_2,H_2PtCl_6: orange-red cryst. from H_2O. M.p. 182°.

Picrolonate: yellow plates from EtOH. M.p. 260°.

N-*Me*: present in tobacco leaves. Oil with ammoniacal odour. B.p. 79–80°. Misc. with H_2O. *B,$HAuCl_4$*: yellow leaflets from dil. HCl. M.p. 190–1°. *Picrolonate*: yellow prisms from H_2O. M.p. 222° decomp. *Methiodide*: cryst. from EtOH. M.p. 286°.

N-*Phenyl*: m.p. 101–2°.

N-2 : 4-*Dinitrophenyl*: yellow needles from EtOH. M.p. 124·5°.

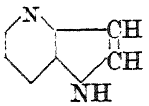
N-*Benzyl*: oil. B.p. about 150°.

N-*Benzoyl*: oil. B.p. 160–1°/2 mm.

N-*Nitroso*: needles from pet. ether. M.p. 37–8°.

Treibs, Dinelli, *Ann.*, 1935, **517**, 172.
Ciamician, Dennstedt, *Ber.*, 1883, **16**, 1536.
Knorr, Rabe, *Ber.*, 1901, **34**, 3497.
Dennstedt, D.R.P., 127,086, (*Chem. Zentr.*, 1902, I, 338).

Pyrrolo-[3, 2-b]-pyridine (4-*Azaindole*)



$C_7H_6N_2$ MW, 118

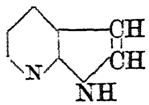
Colourless prisms. M.p. 127–8°. Sol. $CHCl_3$, C_6H_6. Spar. sol. ligroin.

Picrate: bright yellow plates. M.p. 242–4° decomp.

Benzoyl: colourless needles. M.p. 92–6°.

Clemo, Swan, *J. Chem. Soc.*, 1948, 198.

Pyrrolo-[2, 3-b]-pyridine (7-*Azaindole*)



$C_7H_6N_2$ MW, 118

Colourless prisms from C_6H_6–ligroin. M.p. 106–7°.

Picrate: bright yellow prisms from Me_2CO. M.p. 232–3°.

Acetyl: colourless cryst. from ligroin. M.p. 64°.

Clemo, Swan, *J. Chem. Soc.*, 1945, 603.

Pyrrone.

See 2 : 2′-Dipyrryl Ketone.

Pyrrotriazole.

See 1 : 2 : 3 : 4-Tetrazole.

1-Pyrrylisopropyl Alcohol.

See 2-β-Hydroxypropylpyrrole.

Pyruvic Acid (1-*Ketopropionic acid*, *pyroracemic acid*)

CH_3·CO·COOH

$C_3H_4O_3$ MW, 88

Liq. with odour resembling acetic acid. M.p. about 13·6°. B.p. 165° part. decomp., 75–80°/25 mm., 65°/10 mm. $D_4^{15·3}$ 1·2668. $n_D^{15·3}$ 1·43025. Misc. in all proportions with H_2O, EtOH, Et_2O. Scratched with Pt wire ⟶ a trimer, m.p. 92°. Warm conc. HNO_3 ⟶ oxalic acid. CrO_3 ⟶ acetic acid. Reduces NH_3.$AgNO_3$ and $HgCl_2$. $k = 3·2 \times 10^{-3}$ at 25°.

Oxime: 1-isonitrosopropionic acid. Decomp. at 180–1° (177°). $k = 5{\cdot}14\ (4{\cdot}7) \times 10^{-4}$ at 25°. *Acetate*: cryst. M.p. 60° decomp.

Semicarbazone: needles from H_2O. M.p. about 200° decomp.

Phenylhydrazone: needles from H_2O. M.p. 192°.

o-*Nitrophenylhydrazone*: yellow needles from EtOH. M.p. 221°.

p-*Nitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 220°.

2 : 4-*Dinitrophenylhydrazone*: yellow cryst. from AcOH. M.p. 218° (213°).

p-*Bromophenylhydrazone*: yellow needles from AcOH. M.p. 184°.

m-*Nitrobenzoylhydrazone*: m.p. 185·5–186·5°.

Me ester: $C_4H_6O_3$. MW, 102. B.p. 134–7°. D^0 1·154. *Oxime*: needles from Et_2O. M.p. 69°. B.p. 122–3°/14 mm. *Oxime acetate*: m.p. 42°. B.p. 126°/14 mm. 2 : 4-*Dinitrophenylhydrazone*: m.p. 186·5–187·5°. *Di-Me acetal*: b.p. 66–66·5°/16 mm. $D_4^{17\cdot6}$ 1·0678. $n_D^{17\cdot6}$ 1·412.

Et ester: $C_5H_8O_3$. MW, 116. B.p. 155° (144°), 55°/17 mm. $D_4^{15\cdot6}$ 1·0596. $n_D^{15\cdot6}$ 1·408. *Oxime*: prisms or needles. M.p. 97°. B.p. 213° slight decomp. *Semicarbazone*: m.p. 206° decomp. *Phenylhydrazone*: two forms. (i) M.p. 117–18°. (ii) M.p. 31–2°. p-*Nitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 187°. 2 : 4-*Dinitrophenylhydrazone*: yellow cryst. from dioxan–EtOH. M.p. 154·5–155°. *Di-Et acetal*: b.p. 190–1° slight decomp., 81·5–82·5°/15 mm. $D_4^{18\cdot2}$ 0·9783. $n_D^{17\cdot1}$ 1·415.

Propyl ester: $C_6H_{10}O_3$. MW, 130. B.p. 166°. *Semicarbazone*: m.p. 178°. 2 : 4-*Dinitrophenylhydrazone*: m.p. 119–20°.

Butyl ester: $C_7H_{12}O_3$. MW, 142. B.p. 70–1°/11 mm.

d-*Amyl ester*: $C_8H_{14}O_3$. MW, 158. B.p. 185–6°. 85–6°/16 mm., 81–2°/10 mm. D_4^{18} 0·9724. n_D^{18} 1·4206. $[\alpha]_D^{15} + 3{\cdot}25°$.

Isoamyl ester: b.p. 185°, 86°/14 mm. D^{17} 0·978.

Hexadecyl ester: $C_{19}H_{36}O_3$. MW, 312. M.p. 26·5–27·5°. *Semicarbazone*: m.p. 140–1°.

Allyl ester: $C_6H_8O_3$. MW, 128. B.p. 165°, 65°/14 mm. $D_4^{17\cdot5}$ 1·082.

Cyclohexyl ester: $C_9H_{14}O_3$. MW, 170. B.p. 78–80°/3 mm.

l-*Menthyl ester*: oil. B.p. 136–40°/22 mm., 131–2°/10 mm. $D_4^{19\cdot6}$ 0·9852. $[\alpha]_D^{20} - 82{\cdot}00°$ in $CHCl_3$.

Benzyl ester: b.p. 207–8°, 103–4°/36 mm. D^{14} 1·090. *Semicarbazone*: m.p. 176°.

Amide: $C_3H_5O_2N$. MW, 87. Prisms or plates from EtOH. M.p. 124–5°. Sol. H_2O, $CHCl_3$, hot C_6H_6. *Oxime*: plates or prisms from H_2O. M.p. 178·5° decomp. *Semicarbazone*: m.p. 230° decomp.

N-*Carboxymethylamide*: colourless cryst. M.p. 90°.

Nitrile: acetyl cyanide. C_3H_3ON. MW, 69. Liq. with characteristic odour. B.p. 93°. D_4^{20} 0·9745. n^{20} 1·3743. *Phenylhydrazone*: leaflets from C_6H_6. M.p. 150–1°. *Semicarbazone*: m.p. 215° decomp.

Anil: 1-phenyliminopropionic acid, anilpyruvic acid. M.p. 127–8° decomp. Sol. H_2O, EtOH. Spar. sol. AcOEt, C_6H_6. Very spar. sol. $CHCl_3$. Sol. conc. H_2SO_4 with wine-red col.

o-*Tolylimide*: m.p. 146° decomp.

p-*Tolylimide*: m.p. 127°.

Anilide: needles from H_2O or EtOH. M.p. 104°. Sublimes. *Oxime*: yellowish plates from EtOH. M.p. 119°.

N-*Me-anilide*: cryst. from H_2O. M.p. 152–3°.

o-*Toluidide*: needles from EtOH. M.p. 70–1°.

p-*Toluidide*: plates from C_6H_6. M.p. 109°. *Oxime*: leaflets from EtOH. M.p. 130°.

Howard, Fraser, *Organic Syntheses*, Collective Vol. I, 462.
Fernbach, Strange, B.P., 14,607, (*Chem. Abstracts*, 1919, **13**, 1595).
Boehringer Sohn, D.R.P., 523,190, (*Chem. Zentr.*, 1931, II, 496).
Tschelinzeff, Schmidt, *Ber.*, 1929, **62**, 2211.

Pyruvic Alcohol.

See Hydroxyacetone.

Pyruvic Aldehyde (1-*Ketopropionaldehyde, methylglyoxal, pyroracemic aldehyde, propanalone*)

$CH_3{\cdot}CO{\cdot}CHO$

$C_3H_4O_2$ MW, 72

Yellow liq. with pungent odour. Begins to boil at 72° ⟶ a yellowish-green vapour. Liq. at room temp. is bimolecular, rapidly polymerising to an amorphous glassy mass of unknown MW. Latter at 50° ⟶ bimolecular liq. or monomolecular vap.

Oxime: *see* Isonitrosoacetone.

Dioxime: *see* Methylglyoxime.

Phenylosazone: m.p. 145° (154·8°).

Disemicarbazone: m.p. 254°.

o-*Nitrophenylhydrazone*: yellow needles from EtOH. M.p. 128°.

m-*Nitrophenylhydrazone*: pale yellow needles from EtOH. M.p. 152°.

p-*Nitrophenylhydrazone*: yellow needles from EtOH. M.p. 217°.

Di-p-*nitrophenylhydrazone*: m.p. 302–4°.

Di-2 : 4-*dinitrophenylhydrazone*: reddish-orange cryst. from $PhNO_2$. M.p. 299–300°.

Di-m-*nitrobenzoylhydrazone*: m.p. 288·5°.

Dihydrazone: cryst. from EtOH. M.p. 93–4°.

Di-Me acetal: b.p. 143–7°.

Di-Et acetal: liq. with odour resembling acetone. B.p. 161·7–161·8°/761 mm., 54–5°/13–15 mm.

Di-isopropyl acetal: b.p. 92·5°/50 mm. D^{20} 0·900. n^{20} 1·4034.

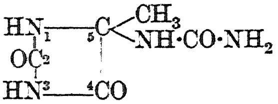
Dibutyl acetal: b.p. 103°/10 mm. D^{20} 0·905. n^{20} 1·4180.

Tetra-Et acetal: b.p. 192°.

Diacetate: pale yellow liq. B.p. 115–16°/13 mm.

Meisenheimer, *Ber.*, 1912, **45**, 2637.
Neuberg, Färber, Levite, Schwenk, *Biochem. Z.*, 1917, **83**, 264.
Dakin, Dudley, *J. Biol. Chem.*, 1913, **15**, 130.
Neuberg, Dalmer, *Biochem. Z.*, 1925, **162**, 488.
Fischer, Taube, *Ber.*, 1924, **57**, 1506.
Henze, Müller, *Z. physiol. Chem.*, 1933, **214**, 281.

Pyruvil (5-*Methylallantoin*)



$C_5H_8O_3N_4$ MW, 172

Plates from H_2O. Insol. EtOH, Et_2O. Heat of comb. 567·7 Cal.

Davidson, *J. Am. Chem. Soc.*, 1925, **47**, 255.
Simon, *Compt. rend.*, 1901, **133**, 587.

Pyruvylformic Acid.

See Diketobutyric Acid.

Q

Quassin.

Bitter constituent of quassia or Surinam wood (*Quassia amara*) and of *Aeschrion excelsa*. Molecular complex of picrasmin (isoquassin) and neoquassin. Rods or plates from MeOH.Aq. M.p. 205–6°. $[\alpha]_D^{20} + 39{\cdot}8°$ in $CHCl_3$.

Clark, *J. Am. Chem. Soc.*, 1937, **59**, 927, 2511; 1938, **60**, 1146.
Adams, Whaley, *J. Am. Chem. Soc.*, 1950, **72**, 375.

***p*-Quaterphenyl.**

See Benzerythrene.

Quebrachamine (*Kamassine*)

$C_{19}H_{26}N_2$ MW, 282

Isolated from quebracho bark. Leaflets from EtOH or C_6H_6. M.p. 147°. $[\alpha]_D - 109{\cdot}5°$ in Me_2CO. Weak tertiary base. Sol. Me_2CO. Mod. sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

$B,H_2SO_4,2H_2O$: prisms. Spar. sol. cold H_2O.
$B,(COOH)_2$: prisms from EtOH. M.p. 217°.
Picrate: scarlet needles from EtOH. M.p. 195–6°.
Methiodide: cream-coloured prisms from MeOH. M.p. 234°. Sol. EtOH, Me_2CO. Insol. Et_2O, C_6H_6.
Methosulphate: prisms. M.p. 235°. Sol. EtOH. Mod. sol. H_2O.

Hesse, *Ann.*, 1882, **211**, 249.
Field, *J. Chem. Soc.*, 1924, **125**, 1444.

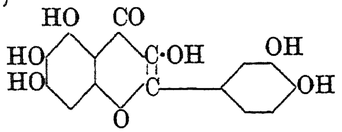
Quebrachine.

See Yohimbine.

Quebrachitol.

See under Inositol.

Quercetagetin (3 : 5 : 6 : 7 : 3′ : 4′-*Hexahydroxyflavone*)



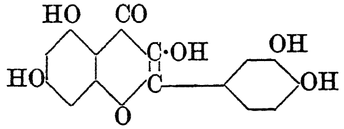
$C_{15}H_{10}O_8$ MW, 318

Isolated from flowers of African marigold (*Tagetes patula*). Cryst. + $2H_2O$ from dil. EtOH. Pale yellow needles or leaflets. M.p. 318–20°. Sol. hot EtOH, dil. alkalis. Spar. sol. boiling H_2O. Ox. by air becoming olive-green and finally brown. $FeCl_3$ in EtOH ⟶ olive-green col. Lead acetate in EtOH ⟶ orange-red ppt. KOH fusion ⟶ protocatechuic acid. Forms oxonium salts with min. acids.

$C_{15}H_{10}O_8,H_2SO_4$: orange needles. Hyd. by H_2O.
Hexa-acetyl: needles from EtOH–AcOH. M.p. 209–11° (203–5°).
6-*Me ether*: *see* Patuletin.
Tetra-Me ether: m.p. 75–6°. *Acetyl*: m.p. 209–10°.
Penta-Me ether: pale yellow needles from EtOH. M.p. 161–2°. *Acetyl*: m.p. 161–3°.
Hexa-Me ether: colourless needles from Me_2CO. M.p. 157–8° (140–1°).
Hexa-Et ether: cryst. from EtOH. M.p. 139–41°.

Latour, Magnier de la Source, *Bull. soc. chim.*, 1877, **28**, 337.
Perkin, *J. Chem. Soc.*, 1913, **103**, 209.
Baker, Nodzu, Robinson, *J. Chem. Soc.*, 1929, 74.
Mahal, *J. Indian Chem. Soc.*, 1938, **15**, 87, (*Chem. Abstracts*, 1938, **32**, 7514).
Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23*A***, 140, (*Chem. Abstracts*, 1947, **41**, 121).

Quercetin (*Meletin, sophoretin, quercitin*, 3 : 5 : 7 : 3′ : 4′-*pentahydroxyflavone*)



$C_{15}H_{10}O_7$ MW, 302

Found in rind of many fruits. Yellow needles + $2H_2O$ from EtOH.Aq. M.p. anhyd. 313–14°. Very sol. aq. alkalis with golden-yellow col. Sol. AcOH, boiling EtOH. Spar. sol. hot H_2O. Conc. H_2SO_4 ⟶ yellow sol. with faint green fluor. Reduces $NH_3.AgNO_3$ in cold and Fehling's on heating. Tasteless.

5-*Me ether*: $C_{16}H_{12}O_7$. MW, 316. Prismatic needles from EtOH. M.p. 305–8°. Alkalis ⟶ orange sols. *Tetra-acetyl*: needles from Me_2CO. M.p. 202–4°.
7-*Me ether*: *see* Rhamnetin.
3′-*Me ether*: *see* Isorhamnetin.
4′-*Me ether*: m.p. 260°. *Tetra-acetyl*: m.p. 165°.
7 : 3′-*Di-Me ether*: *see* Rhamnazin.
3 : 3′ : 4′-*Tri-Me ether*: $C_{18}H_{16}O_7$. MW, 344. Pale yellow needles from AcOEt. M.p. 240–5°. Alc. $FeCl_3$ ⟶ intense greenish-brown col. Conc. H_2SO_4 ⟶ yellow sol. with weak green fluor. *Diacetyl*: needles from EtOH. M.p. 159–60°.
3 : 5 : 3′ : 4′-*Tetra-Me ether*: $C_{19}H_{18}O_7$. MW, 358. Needles from EtOH. M.p. 284–5°. Spar. sol. EtOH. Alkalis ⟶ pale yellow sols. *Acetyl*: needles. M.p. 174–6°.
3 : 7 : 3′ : 4′-*Tetra-Me ether*: pale yellow needles from EtOH. M.p. 159–60° (155°).

Spar. sol. EtOH. Alc. KOH ⟶ bright yellow col. *Acetyl*: needles from EtOH. M.p. 169–70°. Spar. sol. cold EtOH.

5 : 7 : 3′ : 4′-*Tetra-Me ether*: needles from EtOH. M.p. 197–8°. Conc. H_2SO_4 ⟶ yellow sol. with green fluor. *Acetyl*: needles from EtOH. M.p. 160–3°.

Penta-Me ether: $C_{20}H_{20}O_7$. MW, 372. Needles from EtOH or AcOEt. M.p. 151–2°. Gives no col. with alc. KOH.

7 : 3′ : 4′-*Tri-Et ether*: $C_{21}H_{22}O_7$. MW, 386. Yellow needles from EtOH. M.p. 123–4°.

3 : 7 : 3′ : 4′-*Tetra-Et ether*: $C_{23}H_{26}O_7$. MW, 414. Yellow needles. M.p. 121–2°. Mod. sol. cold EtOH. Insol. H_2O. *Acetyl*: needles from 70% EtOH. M.p. 152–3°. Mod. sol. cold EtOH. Dil. alc. sol. shows faint blue fluor.

Penta-Et ether: $C_{25}H_{30}O_7$. MW, 442. Needles. M.p. 116–18°.

3 : 7 : 3′ : 4′-*Tetra-acetyl*: needles. M.p. 193–4°.

Penta-acetyl: needles from EtOH or C_6H_6. M.p. 193·5° (190–1°).

Pentachloroacetyl: needles. M.p. 180°.

Pentabenzoyl: needles from hot Me_2CO. M.p. 188–90°.

3-*Glucoside*: *see* Isoquercitrin.

7-*Glucoside*: *see* Quercimeritrin.

3-*Rhamnoside*: *see* Quercitrin.

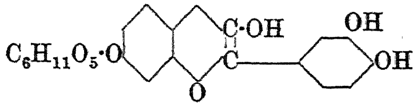
3-*Galactoside*: *see* Hyperin.

3-*Rutinoside*: *see* Rutin.

?-*Glucoside*: incarnatrin. $C_{21}H_{20}O_{12}$. MW, 464. In flowers of *Trifolium incarnatum*, Linn. Yellow needles + $3H_2O$ from H_2O. Softens at 165°, decomp. at 242–5°. Position of glucose not known.

Perkin, Pate, *J. Chem. Soc.*, 1895, **67**, 646.
v. Kostanecki, Lampe, Tambor, *Ber.*, 1904, **37**, 1404.
Herzig, *Monatsh.*, 1888, **9**, 541; 1912, **33**, 690.
Attree, Perkin, *J. Chem. Soc.*, 1927, 239.
Allan, Robinson, *J. Chem. Soc.*, 1926, 2336.
Kubota, Perkin, *J. Chem. Soc.*, 1925, 1894.
Rogerson, *J. Chem. Soc.*, 1910, **97**, 1004.

Quercimeritrin (*Quercetin 7-glucoside*)



$C_{21}H_{20}O_{12}$ MW, 464

Isolated from cotton flowers, leaves of *Gossypium hirsutum, Helianthus annus*, and from bark of *Prunus seratina*, Ehrh. Yellow plates. M.p. 247–9°. Yellow needles + $3H_2O$ from Aq.Py. M.p. 246–8°. Loses H_2O at 100°. Prac. insol. hot H_2O. Hyd. ⟶ quercetin + glucose.

Octa-acetyl: m.p. 216–17° (214–16°).

Penta-Me ether: cryst. + $2H_2O$. Sinters at 197°. M.p. 203–5°.

Perkin, *J. Chem. Soc.*, 1909, **96**, 2185.
Sando, *J. Biol. Chem.*, 1925, **64**, 71; 1926, **68**, 407.
Attree, Perkin, *J. Chem. Soc.*, 1927, 237.
Neelakantan, Rao, Seshadri, *Chem. Zentr.*, 1936, I, 3518.

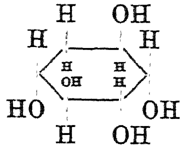
Quercin.
See Scyllitol.

Quercitin.
See Quercetin.

Quercitol (*Pentahydrocyclohexane*)

$C_6H_{12}O_5$ MW, 164

Proto*quercitol* (d-*Quercitol*)



Occurs in acorns, oak bark, leaves of *Chamærops humilis*, Linn., seeds of *Mimusops elengi*, and *Achras sapota*, Linn. Prisms from H_2O or dil. EtOH. M.p. 234° (232°, 235–7°). Sol. H_2O. Insol. cold EtOH, Et_2O. $[\alpha]_D^{15}$ + 24·37° in H_2O, $[\alpha]_D^{20}$ + 25·6° in H_2O. Non-fermentable. HNO_3 ⟶ mucic acid. $KMnO_4$ ⟶ oxalic and malonic acids. *d*- and *l*-quercitol are not optical antipodes.

Monoacetyl: cryst. Sol. Et_2O.

Diacetyl: hard friable mass. Sol. EtOH.

Triacetyl: amorph. Sol. EtOH, Et_2O. Insol. H_2O.

Tetra-acetyl: amorph. brittle mass. Hygroscopic.

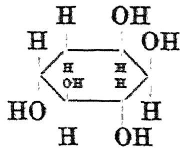
Penta-acetyl: amorph. Sol. Et_2O. Mod. sol. EtOH. Spar. sol. H_2O.

Pentabenzoyl: m.p. 155°. $[\alpha]_D^{23}$ + 61·4°.

Pentacarbanilate: amorph. M.p. 120–40°. Insol. ligroin.

Prunier, *Ann. chim. phys.*, 1878, **15**, 9.
Kiliani, Schäfer, *Ber.*, 1896, **29**, 1762.
Missenden, *Chem. News*, 1922, **125**, 120.
Karrer, *Helv. Chim. Acta*, 1926, **9**, 116.
Kiliani, *Ber.*, 1931, **64**, 2473.
Posternak, *Helv. Chim. Acta*, 1932, **15**, 948.

Vibo*quercitol* (*Viburnitol*, "l-*quercitol*")



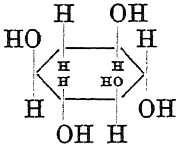
Isolated from leaves of *Viburnum tinus*, Linn., and *Gymnema sylvestre*, Br. Needles + $1H_2O$ from H_2O. M.p. anhyd. 180–1° corr. Very sol. H_2O. Spar. sol. EtOH. $[\alpha]_D$ − 49·5° in H_2O.

Penta-acetyl: needles from dil. EtOH. M.p. 125–6° corr. Cryst. + C_6H_6 from C_6H_6. M.p. 87–97°. $[\alpha]_D$ − 26° in $CHCl_3$.

Pentabenzoyl: amorph. from EtOH, m.p. 133°. Needles + EtOH from pet. ether–AcOEt–EtOH, m.p. 116°. $[\alpha]_D$ − 79° in $CHCl_3$.

Power, Tutin, *J. Chem. Soc.*, 1904, **85**, 624.
Hérissey, Poirot, *J. pharm. chim.*, 1937, **26**, 385, (*Chem. Abstracts*, 1938, **32**, 7024).
Posternak, Schopfer, *Helv. Chim. Acta*, 1950, **33**, 343.
Posternak, *ibid.*, 350.

Scyllo*quercitol* (" *Deoxscyllitol* ")



Penta-acetyl: cryst. from EtOH. M.p. 188–90°.

Posternak, *Helv. Chim. Acta*, 1941, **24**, 1045; 1950, **33**, 350.

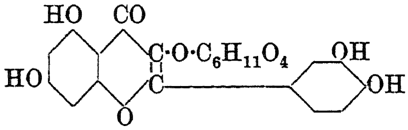
dl-Epi*quercitol*.
Prisms from EtOH.Aq. M.p. 206–8°.

May, Mosettig, *J. Org. Chem.*, 1949, **14**, 1137.

General reference.
Angyal, Macdonald, Matheson, *J. Chem. Soc.*, 1952, 686.

l-Quercitol.
See under Quercitol.

Quercitrin (*Quercetin 3-rhamnoside*)



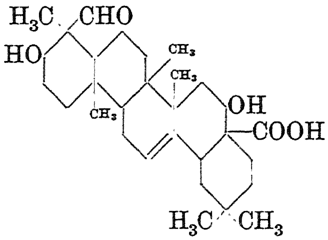
$C_{21}H_{20}O_{11}$ MW, 448

Occurs in quercitron bark. Pale yellow leaflets + $2H_2O$. M.p. 182–5° (air dried), 250–2° anhyd. Sol. EtOH. Insol. cold H_2O. Gives yellow ppt. with aq. lead acetate. Hyd. ⟶ quercetin + rhamnose.

Hepta-acetyl: powder. $[\alpha]_D^{24}$ − 165·6° in $CHCl_3$. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6. Insol. H_2O, Et_2O, pet. ether.

Zwenger, Dronke, *Ann.*, *Suppl.*, 1861, **1**, 267.
Liebermann, Hamburger, *Ber.*, 1879, **12**, 1179.
Zemplén, Csürös, Gerecs, Aczél, *Ber.*, 1928, **61**, 2486.

Quillaic Acid



$C_{30}H_{46}O_5$ MW, 486

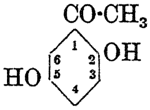
Sapogenin obtained on hydrolysis of quillaia saponin. Needles from EtOH. M.p. 294°. $[\alpha]_D$ + 56·1° in Py. Tetranitromethane ⟶ yellow col.

Me ester: $C_{31}H_{48}O_5$. MW, 500. Needles from MeOH. M.p. 225°. $[\alpha]_D$ + 40·5° in Py. *Oxime*: needles. M.p. 238°.

Diacetyl: needles from AcOH. M.p. 250°.
Oxime: needles from MeOH. M.p. 282°.
Semicarbazone: needles. M.p. 288°.

Windaus, Hampe, Rabe, *Z. physiol. Chem.*, 1926, **160**, 301.
Elliott, Kon, *J. Chem. Soc.*, 1939, 1130.
Bilham, Kon, *J. Chem. Soc.*, 1940, 1471; 1941, 552.
Bilham, Kon, Ross, *J. Chem. Soc.*, 1942, 532.
Bischof, Jeger, Ruzicka, *Helv. Chim. Acta*, 1949, **32**, 1911.

Quinacetophenone (*Acetohydroquinone*, 2:5-*dihydroxyacetophenone*)



$C_8H_8O_3$ MW, 152

Yellowish-green cryst. from H_2O. M.p. 202°. Sol. EtOH. Spar. sol. Et_2O, C_6H_6. Prac. insol. cold H_2O. Sol. alkalis with yellow col. $FeCl_3$ ⟶ transient blue col. Sublimes.

5-*Me ether*: $C_9H_{10}O_3$. MW, 166. Yellow prisms from dil. EtOH. M.p. 52°. *Benzoyl*: m.p. 55–7°. p-*Nitrobenzoyl*: m.p. 146–7°. 3:5-*Dinitrobenzoyl*: m.p. 139–40°.

2:5-*Di-Me ether*: $C_{10}H_{12}O_3$. MW, 180. Cryst. M.p. 20–2°. B.p. 155–8°/14 mm. D_4^{20} 1·1385. *Semicarbazone*: needles from dil. EtOH. M.p. 181–2°. *Phenylhydrazone*: yellow prisms from EtOH. M.p. 99–100°.

5-*Et ether*: $C_{10}H_{12}O_3$. MW, 180. Yellow prisms from EtOH. M.p. 57°.

2:5-*Di-Et ether*: $C_{12}H_{16}O_3$. MW, 208. Cryst. from EtOH. M.p. 42°.

5(?)-*Acetyl*: yellow needles from AcOH. M.p. 91°. *Phenylhydrazone*: needles from dil. EtOH or C_6H_6. M.p. 147°.

2:5-*Diacetyl*: needles from AcOH. M.p. 68°.

2:5-*Dibenzoyl*: prisms from EtOH–C_6H_6.

M.p. 113°. *Phenylhydrazone*: yellow needles from dil. EtOH. M.p. 148°.

Oxime: plates from toluene. M.p. 149–50°.

p-*Nitrophenylhydrazone*: m.p. 215–16°.

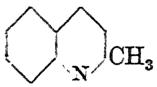
Nencki, Schmid, *J. prakt. Chem.*, 1881, **23**, 546.
Klinger, Kolvenbach, *Ber.*, 1898, **31**, 1214.
Kostanecki, Lampe, *Ber.*, 1904, **37**, 774 (*Footnote*).
Kauffmann, Beisswenger, *Ber.*, 1905, **38**, 791.
Amin, Shah, *Organic Syntheses*, 1948, XXVIII, 42.
Oliverio, Lugli, *Gazz. chim. ital.*, 1948, **78**, 16.

Quinacrine.
See Atebrin.

Quinaform.
See under Quinine.

Quinaldic Acid.
See Quinaldinic Acid.

Quinaldine (2-*Methylquinoline*)



$C_{10}H_9N$ MW, 143

Present in coal tar. F.p. $-2°$ to $-1°$. B.p. 247·6°/760 mm., 238–9°/716 mm., 135·5°/26 mm., 118°/10 mm. D_4^{20} 1·0585. n_D^{20} 1·6126. Heat of comb. C_v 1286·27 Cal. $k = 3{\cdot}6 \times 10^{-9}$ at 14°, $7{\cdot}4 \times 10^{-9}$ at 25°. $CrO_3 \longrightarrow$ quinaldinic acid. $KMnO_4 \longrightarrow$ acetylanthranilic acid. Sn + HCl $\longrightarrow$ 1 : 2 : 3 : 4-tetrahydroquinaldine. Conc. $HNO_3 \longrightarrow$ nitroquinaldinic acid. $HNO_3 + H_2SO_4 \longrightarrow$ 5- and 8-nitroquinaldines. $H_2SO_4 \longrightarrow$ quinaldine-5-, -6-, and -8-sulphonic acids. $SeO_2 \longrightarrow$ quinoline-2-aldehyde and quinaldinic acid.

B,HCl: needles from EtOH–Et_2O. M.p. 224°.

B,2HCl: m.p. about 31°.

B_2,HBr: cryst. + $3H_2O$. M.p. 54°.

B,HI: needles from EtOH. M.p. 186°. Sol. H_2O.

B,H_2SO_4: prisms. M.p. 211–13°.

B_2,H_2PtCl_6: orange-red prisms from H_2O. M.p. 228–9° (226°, 230–41°). Less sol. hot H_2O than quinoline chloroplatinate.

$B_2,H_2Cr_2O_7$: yellowish-red needles. Sol. hot H_2O. Also yellow needles + $3H_2O$ for H_2O. M.p. 110°.

$B_2,2HCl,ZnCl_2$: decomp. at 245°.

$B,HCl,HgCl_2$: needles. M.p. 165·5°.

$B_2,2HCl,CuCl_2$: orange tablets. M.p. 175–8° decomp.

Picrate: yellow needles. M.p. 194° (191°). Spar. sol. H_2O, cold EtOH.

Styphnate: m.p. 213–14°.

N-*Oxide*: needles + H_2O (or $\frac{1}{2}H_2O$) from H_2O. M.p. 77–8°. Decomp. at 200°. Sol. most org. solvents and acids. Insol. alkalis. B_4,H_2PtCl_6: yellow prisms + $2H_2O$. M.p. 207° decomp. *Picrate*: yellow cryst. from EtOH. M.p. about 173°.

Methiodide: yellow needles from EtOH. M.p. 195°. Sol. H_2O. Insol. Et_2O.

Ethiodide: yellow needles from EtOH. M.p. 233–4°. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O.

Propiodide: greenish-yellow prisms from EtOH. M.p. 166–7° decomp.

Isobutyliodide: yellow plates from EtOH. M.p. 172°.

Isoamyliodide: yellow prisms from EtOH. M.p. 175°. Sol. H_2O. Spar. sol. cold EtOH.

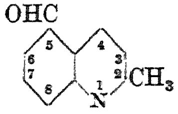
Methyl-p-*toluenesulphonate*: cryst. M.p. 134°.

Ethyl-benzenesulphonate: cryst. M.p. 105°.

Methoperchlorate: prisms from MeOH. M.p. 154°. Mod. sol. cold H_2O.

Doebner, v. Miller, *Ber.*, 1883, **16**, 2465.
Skraup, *Ber.*, 1882, **15**, 897.
Schering, D.R.P., 24,317.
Mills, Harris, Lambourne, *J. Chem. Soc.*, 1921, **119**, 1297.
Basu, *Ann.*, 1934, **512**, 134.
Rabinovich, Dzirkal, *Chem. Abstracts*, 1934, **28**, 3725.
Tseou Heou-Feo, *Bull. soc. chim.*, 1935, 2, 90.

Quinaldine-5-aldehyde (2-*Methylquinoline-5-aldehyde, 5-aldehydoquinaldine*)



$C_{11}H_9ON$ MW, 171

Cryst. + H_2O from H_2O. M.p. 73°, anhyd. 61°. Sol. EtOH, Et_2O, Me_2CO, C_6H_6, dil. min. acids. Spar. sol. hot H_2O, pet. ether. Volatile in steam. $Ag_2O \longrightarrow$ quinaldine-5-carboxylic acid.

B_2,H_2PtCl_6: orange-yellow plates from alc. HCl. M.p. 211°. Spar. sol. hot EtOH.

Picrate: needles from EtOH. M.p. 182° decomp.

Eckhardt, *Ber.*, 1889, **22**, 277.
See also Decker, Remfry, *Ber.*, 1905, **38**, 2775.

Quinaldine-6-aldehyde (2-*Methylquinoline-6-aldehyde, 6-aldehydoquinaldine*).

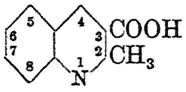
Needles from H_2O, plates from C_6H_6–pet. ether, m.p. 106°. Sol. EtOH, Et_2O, C_6H_6, min. acids. Mod. sol. hot H_2O, pet. ether.

Phenylhydrazone: golden-yellow prisms from EtOH. M.p. 160°.

B_2,H_2PtCl_6: orange prisms + $2H_2O$.

v. Miller, Kinkelin, *Ber.*, 1885, **18**, 3237.

Quinaldine-3-carboxylic Acid (2-*Methylquinoline*-3-*carboxylic acid*)



$C_{11}H_9O_2N$ MW, 187

Needles from EtOH or C_6H_6. M.p. 238° (235° decomp., 251° decomp.). Spar. sol. most solvents. Prac. insol. H_2O. Heat ⟶ quinaldine.

Me ester: $C_{12}H_{11}O_2N$. MW, 201. Needles from EtOH. M.p. 72°. *Methiodide*: yellow needles or plates from H_2O. M.p. 200° decomp. Sol. H_2O, hot EtOH. Insol. Et_2O. *Ethochloride*: needles from EtOH–Et_2O. M.p. 150° decomp. *Ethobromide*: prisms from H_2O. M.p. 154°. *Ethiodide*: yellow plates from H_2O or EtOH. M.p. 210° decomp.

Et ester: $C_{13}H_{13}O_2N$. MW, 215. Needles from dil. EtOH. M.p. 71°. *Methochloride*: yellow plates or needles from H_2O or EtOH. M.p. 158° decomp. *Methiodide*: yellow plates or needles from H_2O or EtOH. M.p. 208° decomp. Spar. sol. cold H_2O, EtOH. *Ethochloride*: needles from EtOH–Et_2O. M.p. 146° decomp. *Ethobromide*: prisms from H_2O. M.p. 217°. *Ethiodide*: orange needles from EtOH. M.p. 236° decomp.

Propyl ester: $C_{14}H_{15}O_2N$. MW, 229. Prisms or needles from EtOH. M.p. 51°. *Methiodide*: yellow needles. M.p. 186° decomp.

Benzyl ester: $C_{18}H_{15}O_2N$. MW, 277. Prisms. M.p. 82°. *Methiodide*: yellow needles from H_2O. M.p. 172°.

Amide: $C_{11}H_{10}ON_2$. MW, 186. Cryst.

Nitrile: 3-cyanoquinaldine. $C_{11}H_8N_2$. MW, 168. Prisms from H_2O or needles from EtOH. M.p. 131° (125–7°).

Friedländer, Gohring, *Ber.*, 1883, **16**, 1836.
Hantzsch, *Ber.*, 1886, **19**, 37.
Claus, Steinitz, *Ann.*, 1894, **282**, 117 (*Footnote*).
Rohde, *Ber.*, 1889, **22**, 267.
v. Walther, *J. prakt. Chem.*, 1903, **67**, 509.
v. Meyer, *J. prakt. Chem.*, 1914, **90**, 27.

Quinaldine-4-carboxylic Acid (2-*Methylquinoline*-4-*carboxylic acid*, 2-*methylcinchoninic acid*, *aniluvitonic acid*).

Yellow cryst. + $1H_2O$ from hot H_2O. M.p. 244° (242°, 246° decomp.). Sol. hot EtOH, AcOH, dil. acids. Spar. sol. Et_2O, C_6H_6. Insol. $CHCl_3$, pet. ether. Sublimes. Heat ⟶ quinaldine. Alk. $KMnO_4$ ⟶ 6-methylpyridine-2 : 3 : 4-tricarboxylic acid. Acid $KMnO_4$ ⟶ acetylanthranilic acid.

Me ester: m.p. 61–2°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Part. decomp. on dist.

Et ester: prisms from pet. ether. M.p. 77°. Sol. EtOH, Et_2O, Me_2CO, C_6H_6. *Picrate*: yellow needles from EtOH. M.p. 156°. *B_2,H_2PtCl_6*: yellow needles + $2H_2O$. M.p. 203° decomp.

Amide: needles from H_2O. M.p. 239°. Sol. hot EtOH. Spar. sol. cold H_2O, Et_2O. Br + NaOH ⟶ 4-aminoquinaldine. *Picrate*: m.p. 239° (231–2° decomp.).

B_2,H_2PtCl_6: yellow needles + $2H_2O$. M.p. 220° decomp.

Picrate: greenish-yellow needles from EtOH. M.p. 191°.

v. Miller, *Ber.*, 1891, **24**, 1918.
Böttinger, *Ann.*, 1878, **191**, 321.
Simon, *Ann. chim. phys.*, 1896, **9**, 466.
Beyer, *J. prakt. Chem.*, 1886, **33**, 411.
Pfitzinger, *J. prakt. Chem.*, 1886, **33**, 100; 1888, **38**, 582; 1897, **56**, 284.
Bayer, D.R.P., 290,703, (*Chem. Zentr.*, 1916, I, 645).
Knövenagel, Bähr, *Ber.*, 1922, 55, 1927.

Quinaldine-5-carboxylic Acid (2-*Methylquinoline*-5-*carboxylic acid*).

Needles from EtOH. M.p. 285° decomp. Sol. cold NH_3 and warm dil. acids. Mod. sol. EtOH. Prac. insol. H_2O. Insol. Et_2O, $CHCl_3$, C_6H_6, ligroin.

Nitrile: 5-cyanoquinaldine. Needles + $2H_2O$ from H_2O. M.p. 82°, anhyd. 104°. Sol. EtOH, Et_2O, C_6H_6, hot H_2O. Volatile in steam.

Doebner, v. Miller, *Ber.*, 1884, **17**, 941.
Eckhardt, *Ber.*, 1889, **22**, 281.
v. Miller, *Ber.*, 1890, **23**, 2263.
Richard, *ibid.*, 3489.
Rist, *ibid.*, 3486.

Quinaldine-6-carboxylic Acid (2-*Methylquinoline*-6-*carboxylic acid*).

Needles from EtOH. M.p. 261–2° (259°, 256°). Sol. hot EtOH. Spar. sol. hot H_2O. Sublimes.

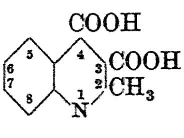
Doebner, v. Miller, *Ber.*, 1884, **17**, 939.
v. Miller, *Ber.*, 1890, **23**, 2263.
I.G., D.R.P., 567,273, (*Chem. Zentr.*, 1933, I, 1687).

Quinaldine-8-carboxylic Acid (2-*Methylquinoline*-8-*carboxylic acid*).

Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. 151°. Sol. H_2O, EtOH, acids, alkalis. Part. decomp. on heating ⟶ quinaldine.

Doebner, v. Miller, *Ber.*, 1884, **17**, 943.
v. Miller, *Ber.*, 1890, **23**, 2259.

Quinaldine-3 : 4-dicarboxylic Acid (2-*Methylquinoline*-3 : 4-*dicarboxylic acid*)



$C_{12}H_9O_4N$ MW, 231

Needles + H_2O from H_2O. M.p. 238–9° (236–7°) (slow heat.), about 245° (rapid heat.). Prac. insol. H_2O.

Di-Me ester: $C_{14}H_{13}O_4N$. MW, 259. M.p. 61–2°. *Picrate*: m.p. 156°.

Di-Et ester: $C_{16}H_{17}O_4N$. MW, 287. M.p. 88–9°. Sol. Et_2O, C_6H_6. Spar. sol. EtOH.

Anhydride: $C_{12}H_7O_3N$. MW, 213. M.p. 218°.

Imide: $C_{12}H_8O_2N_2$. MW, 212. Yellow needles from AcOEt. M.p. 257°.

3-*Nitrile*: 3-cyanoquinaldine-4-carboxylic acid, 2-methyl-3-cyanocinchoninic acid. Plates from EtOH. M.p. 238° decomp. Sol. NaOH. Heat ⟶ 3-cyanoquinaldine.

v. Walther, *J. prakt. Chem.*, 1903, **67**, 504.
Pfitzinger, *J. prakt. Chem.*, 1897, **56**, 316.
Engelhard, *J. prakt. Chem.*, 1898, **57**, 479.
Schering, D.R.P., 275,963, (*Chem. Zentr.*, 1914, II, 182).
Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, **125**, 634.
Mićović, *Bull. soc. chim.*, 1939, **6**, 1156.
Mitchovitch (= Mićović), *Compt. rend.*, 1938, **206**, 1261.

Quinaldine-4 : 6-dicarboxylic Acid (2-*Methylquinoline*-4 : 6-*dicarboxylic acid*).

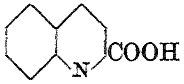
Powder. Sinters at 160°.

v. Miller, *Ber.*, 1890, **23**, 2262.

Quinaldine-sulphonic Acid.

See Methylquinoline-sulphonic Acid.

Quinaldinic Acid (*Quinaldic acid, quinoline*-2-*carboxylic acid*).



$C_{10}H_7O_2N$ MW, 173

Needles + $2H_2O$ from H_2O. Loses H_2O at 100°. M.p. anhyd. 157°. Sol. hot H_2O, C_6H_6. Mod. sol. cold H_2O. *k* (acid) = $1{\cdot}2 \times 10^{-5}$ at 25°. Gives reddish-yellow col. with $FeSO_4$. Heat ⟶ quinoline. Alk. $KMnO_4$ ⟶ pyridine-2 : 3 : 6-tricarboxylic acid. HNO_3 at 60–70° ⟶ 5- and 8-nitroquinaldinic acids. Used in estimation of Zn, Cu, Cd, UO_2, and for separation of Cu from Cd, P, As, Pb, Ni, Co, Mn, etc.

Me ester: $C_{11}H_9O_2N$. MW, 187. Needles from ligroin. M.p. 86° (85°, 78°). *Methiodide*: orange cryst. M.p. 122–9° decomp. *Methochloride*: m.p. 158–65° decomp. *Methonitrate*: m.p. 138° decomp.

Et ester: $C_{12}H_{11}O_2N$. MW, 201. Needles. M.p. 36°.

Nitrile: 2-cyanoquinoline. $C_{10}H_6N_2$. MW, 154. Needles. M.p. 94°. Sol. EtOH, C_6H_6, Et_2O.

Amide: $C_{10}H_8ON_2$. MW, 172. Needles from dil. EtOH. or C_6H_6–ligroin. M.p. 133° (123°). Sol. EtOH, C_6H_6, $CHCl_3$, AcOH. Spar. sol. Et_2O, ligroin, hot H_2O. Sol. dil. HCl.

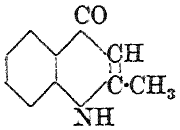
Chloride: $C_{10}H_6ONCl$. MW, 191·5. Exists in two forms. (i) M.p. 175–6°: (ii) m.p. 97°.

N-*Oxide*: needles from H_2O. M.p. 171° decomp. (167° decomp.). Sol. Me_2CO, EtOH, C_6H_6. Spar. sol. Et_2O, ligroin.

Picrate: yellow needles from H_2O. Sol. hot H_2O, EtOH.

Doebner, v. Miller, *Ber.*, 1883, **16**, 2472.
Besthorn, Ibele, *Ber.*, 1906, **39**, 2329.
v. Miller, Krämer, *Ber.*, 1891, **24**, 1915.
Besthorn, *Ber.*, 1909, **42**, 2698.
Meyer, *Monatsh.*, 1904, **25**, 1199.
Mills, Hamer, *J. Chem. Soc.*, 1922, **121**, 2008.
Taylor, *J. Chem. Soc.*, 1929, 1110.
Hammick, *J. Chem. Soc.*, 1923, **123**, 2882.
Hammick, Dickinson, *J. Chem. Soc.*, 1929, 214.
Kaufmann, Dändliker, *Ber.*, 1913, **46**, 2928.

γ-Quinaldone



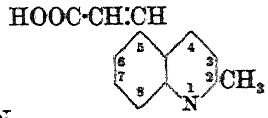
$C_{10}H_9ON$ MW, 159

The compound of the above formula described by Heller and Sourlis (*Ber.*, 1908, **41**, 2696) has been shown by Meisenheimer and Stotz (*Ber.*, 1925, **58**, 2334) to be quinaldine *N*-oxide.

N-*Me*: 1 : 2-dimethyl-γ-quinolone. Needles from C_6H_6. M.p. 176°. Sol. H_2O, EtOH, hot C_6H_6. *B,HCl*: prisms + H_2O. M.p. anhyd. 217°. *B,HI*: needles + H_2O from H_2O. M.p. anhyd. 201°. *B_2,H_2PtCl_6*: yellow cryst. from H_2O. M.p. 240° decomp. Sol. hot H_2O. *Picrate*: decomp. at 233°.

Conrad, Limpach, *Ber.*, 1887, **20**, 956.
Conrad, Eckhardt, *Ber.*, 1889, **22**, 76.
Knorr, *Ber.*, 1897, **30**, 925.

2-[5-Quinaldyl]-acrylic Acid



$C_{13}H_{11}O_2N$ MW, 213

Prisms from EtOH. M.p. 246° decomp. Mod. sol. EtOH, Me_2CO, C_6H_6. Prac. insol. Et_2O, $CHCl_3$, ligroin. $KMnO_4$ ⟶ quinaldine-5-aldehyde.

Ag salt: cryst. + 2 or $4H_2O$.

B,HCl: needles + H_2O.

B,HNO_3: needles + H_2O.

B_2,H_2PtCl_6: yellow plates + $2H_2O$.

Picrate: needles + H_2O from EtOH. M.p.

150–2°. Sol. hot H_2O, EtOH, Me_2CO. Prac. insol. Et_2O.

Eckhardt, *Ber.*, 1889, **22**, 272.
See also Decker, Remfry, *Ber.*, 1905, **38**, 2775.

2-[6-Quinaldyl]-acrylic Acid.
Needles from EtOH. M.p. 240–50°. Spar. sol. cold EtOH. Prac insol. H_2O. Sol. dil. alkalis. Spar. sol. dil. acids. $KMnO_4 \longrightarrow$ quinaldine-6-aldehyde.
B,HCl: prisms + H_2O from dil. HCl. Sol. H_2O.
B,HNO₃: prisms + H_2O. Spar. sol. dil. HNO_3.
B_2,H_2PtCl_6: reddish-yellow prisms + $2H_2O$.

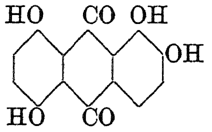
v. Miller, Kinkelin, *Ber.*, 1885, **18**, 3235.

2-[7-Quinaldyl]-acrylic Acid.
Cryst. + ½EtOH from EtOH. M.p. 204°.

Eckhardt, *Ber.*, 1889, **22**, 273.

Quinaldylamine.
See Aminomethylquinoline.

Quinalizarin (1 : 2 : 5 : 8-*Tetrahydroxyanthraquinone*)



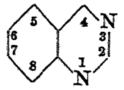
$C_{14}H_8O_6$ MW, 272

Red needles with green metallic lustre from $PhNO_2$. Does not melt below 275°. Very spar. sol. most solvents. Sublimes. Sol. alkalis with reddish-violet col. Sol. conc. H_2SO_4 with bluish-violet col. Zn fusion $\longrightarrow$ anthracene.
Tetra-acetyl: needles from EtOH–$CHCl_3$. M.p. 201°.
1 : 2-*Di-Me ether*: $C_{16}H_{12}O_6$. MW, 300. Brownish-red plates from EtOH or C_6H_6. M.p. 225–30°. Mod. sol. boiling AcOH. Spar. sol. hot EtOH, hot C_6H_6. Sol. alkalis with bluish-violet col. Sol. conc. H_2SO_4 with blue col.

Liebermann, Kostanecki, *Ann.*, 1887, **240**, 301.
Schmidt, *J. prakt. Chem.*, 1891, **43**, 239.
Gattermann, *ibid.*, 249.
Graebe, *Ber.*, 1890, **23**, 3739.

Quinanisole.
See under 6-Hydroxyquinoline *and* 8-Hydroxyquinoline.

Quinazoline (5 : 6-*Benzpyrimidine, phenmiazine*)



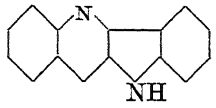
$C_8H_6N_2$ MW, 130

Yellow plates from pet. ether. M.p. 48–48·5°. B.p. 243°/772·5 mm. (241·5°/764 mm.). Sol. H_2O with neutral reaction. Sol. usual solvents. Odour resembles naphthalene. Alk. $KMnO_4 \longrightarrow$ pyrimidine-4 : 5-dicarboxylic acid + 4-hydroxyquinazoline.
$B,HAuCl_4$: orange-red cryst. + H_2O. M.p. 185°.
B_2,H_2PtCl_6: orange-yellow prisms. Does not melt below 250°.
Picrate: needles. M.p. 188–90°.
3-*Methohydroxide*: prisms from H_2O. M.p. 163–5°. Spar. sol. H_2O, Et_2O, C_6H_6, HCl.
3-*Methochloride*: needles. Sinters at 165–6°, m.p. 171–2°. Sol. H_2O.
3-*Methobromide*: m.p. 150–2°. Sol. H_2O.
3-*Methiodide*: yellow prisms + MeOH. M.p. 125–7°. Sol. H_2O.
3-*Ethohydroxide*: m.p. 145–6°. Sol. hot H_2O.
3-*Ethochloride*: m.p. 150–1°. Sol. H_2O.

Gabriel, *Ber.*, 1903, **36**, 808.
Bischer, Lang, *Ber.*, 1895, **28**, 292.
Riedel, D.R.P., 174,941, (*Chem. Zentr.*, 1906, II, 1372).

Quinazolone.
See Hydroxyquinazoline.

Quindoline

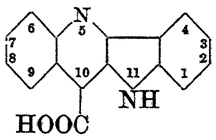


$C_{15}H_{10}N_2$ MW, 218

Needles from EtOH. M.p. 247–8°. Sol. EtOH, Et_2O with blue fluor. Insol. H_2O. Sol. hot dil. HCl, conc. H_2SO_4 with blue fluor. Sublimes with part. decomp.
B,HCl: yellow cryst. Sol. H_2O, EtOH.
B,HI: yellow cryst. Sol. H_2O, EtOH.
B,HNO₃: yellow cryst. Sol. H_2O, EtOH.
Picrate: yellow cryst. Sol. EtOH.
Methochloride: yellow cryst. M.p. 273° decomp.
Methiodide: yellow needles from H_2O.
Methosulphate: orange-yellow needles. M.p. 242–5°.
Ethiodide: yellow needles from H_2O or EtOH. M.p. 222–3°.
Acetyl: yellow needles from EtOH. M.p. 177–8°.

Fichter, Boehringer, *Ber.*, 1906, **39**, 3940.
Fichter, Rohner, *Ber.*, 1910, **43**, 3490.
Noelting, Steuer, *ibid.*, 3512.
Armit, Robinson, *J. Chem. Soc.*, 1922, **121**, 836.

Quindoline-10-carboxylic Acid (*Flavindin*)

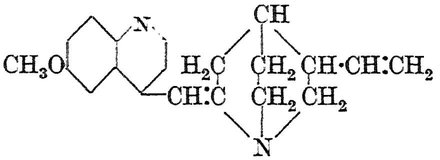


$C_{16}H_{10}O_2N_2$ MW, 262

Yellow ppt. M.p. 336–7° decomp. NaHg or Zn ⟶ quindoline.
Me ester: yellow needles from MeOH.Aq. M.p. 154–5°.
Amide: m.p. 334–6°.

Giraud, *Compt. rend.*, 1879, **89**, 104; 1880, **90**, 1429.
Noelting, Steuer, *Ber.*, 1910, **43**, 3512.
Fichter, Rohner, *ibid.*, 3489.
Armit, Robinson, *J. Chem. Soc.*, 1922, **121**, 836.
Holt, Petrov, *J. Chem. Soc.*, 1947, 607.

Quinene (*Quinenine*)

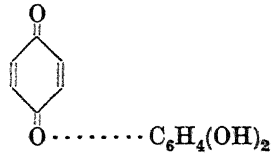


$C_{20}H_{22}ON_2$ MW, 306

Rhombic cryst. + $2H_2O$ from Et_2O or ligroin. Sinters at 75°, m.p. 81–2°. Loses H_2O at 100°. 25% H_3PO_4 at 170–80° ⟶ 6-methoxylepidine + meroquinene. HBr (D 1·49) at 190° ⟶ apoquinene. Br in $CHCl_3$ ⟶ quinene dibromide. Fluoresces in dil. H_2SO_4. Gives thalleioquin reaction.
B,2HCl: yellow needles + H_2O from EtOH. M.p. 180–5°. $[\alpha]_D^{24}$ (anhyd.) + 18·4° in H_2O.
B,2HCl,CuCl₂: yellowish-green cryst. Decomp. at 125–30°.

Comstock, Koenigs, *Ber.*, 1884, **17**, 1989; 1885, **18**, 1223.
Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1920, **42**, 1501.
Cohen, *J. Chem. Soc.*, 1933, 996.

Quinenine.
See Quinene.
Quinethyline.
See under Cupreine.
Quinhydrone (*Molecular comp. of* p-*benzoquinone and hydroquinone*)

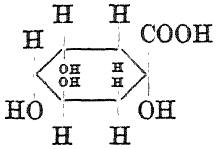


$C_6H_4O_2 + C_6H_6O_2 = C_{12}H_{10}O_4$ MW, 218

Reddish-brown needles with green lustre. M.p. 171°. Sol. EtOH, Et_2O with yellow col. Spar. sol. cold H_2O. Insol. pet. ether, ligroin. Sol. NH_3 with green col. D^{20} 1·401. Sublimes. Boiling H_2O ⟶ quinone + hydroquinone.

Bamberger, Czerkis, *J. prakt. Chem.*, 1903, **68**, 486.
Wöhler, *Ann.*, 1844, **51**, 153.
Torrey, Hardenbergh, *Am. Chem. J.*, 1905, **33**, 168.
Clark, *Am. Chem. J.*, 1892, **14**, 571.
Michael, Cobb, *J. prakt. Chem.*, 1910, **82**, 304.
Evans, Dehn, *J. Am. Chem. Soc.*, 1930, **52**, 3204.
Müller, *Chem. Abstracts*, 1929, **23**, 5471.
Trénel, Bischoff, *Z. angew. Chem.*, 1929, **42**, 288.

Quinic Acid (1 : 3 : 4 : 5-*Tetrahydroxycyclohexane*-1-*carboxylic acid*, 1 : 3 : 4 : 5-*tetrahydroxyhexahydrobenzoic acid*)



$C_7H_{12}O_6$ MW, 192

l-.
Occurs in cinchona bark, coffee beans, bilberries, sugar beet, etc. Prisms from H_2O. M.p. 162° (172°). Sol. 2·5 parts H_2O at 9°. $[\alpha]_D^{15}$ −43·84° in H_2O, $[\alpha]_D^{20}$ − 44·03° in H_2O, $[\alpha]_D^{25\cdot5}$ −42·1° in H_2O. Triboluminescent. Heat at 200–50° ⟶ γ-lactone (quinide). $KMnO_4$ ⟶ *p*-benzoquinone. PbO_2 ⟶ hydroquinone. KOH fusion ⟶ protocatechuic acid. HI at 115–20° ⟶ benzoic acid.
NH₄ salt: cryst. from H_2O. M.p. 179°.
Na salt: prisms or plates + $2H_2O$. Sol. ½ part H_2O at 15°.
Tetra-acetyl: cryst. M.p. 132–6°. Sol. EtOH, Et_2O, alkalis. Mod. sol. H_2O. Spar. sol. CS_2. Insol. ligroin. $[\alpha]_D^{20}$ − 22·50° in EtOH.
Tetrabenzoyl: amorph. + $2H_2O$. M.p. 137–8°. Sol. EtOH, Et_2O. Insol. H_2O.
Me ester: $C_8H_{14}O_6$. MW, 206. Needles from MeOH. M.p. 120° (126° to a milky liquid becoming clear at 142–3°). Sol. H_2O, EtOH. Insol. Et_2O, C_6H_6, pet. ether. *Tetra-Me ether*: m.p. 56–8°. $[\alpha]_D$ − 18·5° in C_6H_6.
Et ester: $C_9H_{16}O_6$. MW, 220. Viscous mass. Sol. H_2O, EtOH. Less sol. Et_2O. *Tetra-acetyl*: plates from H_2O. M.p. 135–6°. Mod. sol. EtOH, Et_2O. Spar. sol. H_2O. Sublimes.
Phenyl ester: $C_{13}H_{16}O_6$. MW, 268. *Tetra-acetyl*: cryst. from dil. EtOH. M.p. 167°. Sol hot EtOH, AcOH. Insol. cold H_2O, Et_2O.
Amide: $C_7H_{13}O_5N$. MW, 191. Cryst. M.p. 132°. Sol. H_2O, EtOH. Insol. Et_2O, C_6H_6. *Tetra-acetyl*: m.p. 186–7° decomp. $[\alpha]_D^{21}$ − 28·6° in tetrachloroethane. *Tetra-Me ether*: m.p. 115–16°.
Nitrile: $C_7H_{11}O_4N$. MW, 173. *Tetra-acetyl*: m.p. 161–2°. $[\alpha]_D^{18}$ − 29·9° in $CHCl_3$.
γ-Lactone: *see* Quinide.

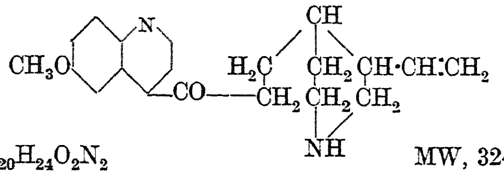
d-.
Prisms. M.p. 164°. Sol. hot H_2O. Spar. sol. EtOH. Prac. insol. Et_2O. $[\alpha]_D^{20}$ + 44° in H_2O.

dl-.

Cryst. $k = 2{\cdot}2 \times 10^{-4}$ at 9°.

Fischer, Dangschat, *Ber.*, 1932, **65**, 1009.
Karrer, Widmer, Riso, *Helv. Chim. Acta*, 1925, **8**, 195.
Herzig, Ortony, *Arch. Pharm.*, 1920, **258**, 91.
Gorter, *Ann.*, 1908, **358**, 329; 1908, **359**, 221.
Erwig, Koenigs, *Ber.*, 1889, **22**, 1461.
Echtermeier, *Arch. Pharm.*, 1906, **244**, 42.
v. Lippemann, *Ber.*, 1901, **34**, 1159.
Hesse, *Ann.*, 1859, **110**, 333.
Zwenger, *Ann.*, 1860, **115**, 108.

Quinicine (*Quinotoxine*)



$C_{20}H_{24}O_2N_2$ MW, 324

One of the cinchona alkaloids. Isomeric with quinine. Reddish-yellow amorph. mass. M.p. about 60°. Sol. EtOH, Et_2O, $CHCl_3$. Prac. insol. H_2O. Sol. in EtOH absorbs CO_2. Forms stable salts. Pptd. from sol. by excess KCNO. $[\alpha]_D^{15}$ + 44·1° in $CHCl_3$. Shows no fluor. in dil. H_2SO_4. Gives thalleioquin reaction.

B,HCl: leaflets. M.p. 180–2° (179–80°). $[\alpha]_D^{13\cdot7}$ + 16·26° in H_2O.

B_2,(COOH)$_2$: prisms + $9H_2O$. M.p. 149°.

Dipicrolonate: m.p. 210°.

Oxime: m.p. 112–16°.

p-*Bromophenylhydrazone*: yellow cryst. M.p. 141°.

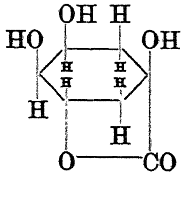
N-*Me*: yellow oil. *B,2HCl*: cryst. + H_2O. M.p. 153–5°. $[\alpha]_D^{23}$ + 16·6° in H_2O.

N-*Et*: yellow oil. *B,HCl*: yellow cryst. from 95% EtOH. M.p. 202–4°. $[\alpha]_D^{23}$ + 68·1° in H_2O.

Dihydride: *see* Quinoticine.

v. Miller, Rohde, Fussenegger, *Ber.*, 1900, **33**, 3228.
Howard, *J. Chem. Soc.*, 1871, **24**, 61; 1872, **25**, 101.
Hesse, *Ann.*, 1875, **178**, 244.
Fränkel, Diamant, *Ber.*, 1925, **58**, 554.
Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1919, **41**, 832; 1922, **44**, 1093.
Proštenik, Prelog, *Helv. Chim. Acta*, 1943, **26**, 1965.

Quinide (*γ-Lactone of quinic acid*)



$C_7H_{10}O_5$ MW, 174

dl-.

Cryst. from H_2O or EtOH. M.p. 200° (198°). Sol. H_2O. Spar. sol. dil. EtOH. Neutral reaction.

l-.

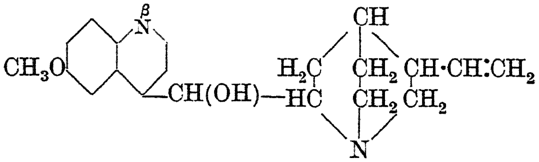
M.p. 187°. $[\alpha]_D^{17}$ − 17·13° in H_2O.

Triacetyl: prisms from EtOH. M.p. 133–4°. $[\alpha]_D^{15}$ − 13·4° in Me_2CO.

Tribenzoyl: cryst. from EtOH. M.p. 148°.

Erwig, Koenigs, *Ber.*, 1889, **22**, 1458.
Hesse, *Ann.*, 1859, **110**, 335.
v. Lippmann, *Ber.*, 1901, **34**, 1159.
Echtermier, *Arch. Pharm.*, 1906, **244**, 53.
Gorter, *Ann.*, 1908, **359**, 223.
Fischer, *Ber.*, 1921, **54**, 781.

Quinidine (*Conquinine, conchinine*)



$C_{20}H_{24}O_2N_2$ MW, 324

One of the cinchona alkaloids. Stereoisomeric with quinine. Cryst. + $2\frac{1}{2}H_2O$ from dil. EtOH. Loses $\frac{1}{2}H_2O$ on exposure to air. Cryst. + EtOH from EtOH. Loses EtOH at 100°. M.p. anhyd. 174–5° (171·5°, 170–1°, 168°). $[\alpha]_D$ + 262° (251°) in EtOH, $[\alpha]_D^{15}$ + 236·8° in 97% EtOH, + 243·5° in 99% EtOH. $[\alpha]_D$ + 230° in $CHCl_3$. Sol. 6900 parts H_2O at 25°, 750 parts at 100°. Sol. 26 parts EtOH, 22 parts Et_2O at 20°. Anhyd. quinidine sol. 4950 parts H_2O, 129 parts Et_2O, 57 parts AcOEt, 177 parts CCl_4, 41 parts C_6H_6 at 18–22°. The salts of quinidine, with the exception of the hydriodide, are more sol. than those of quinine. Quinidine forms add. comps. with alcohols, acetone, benzene, etc. Cold acid $KMnO_4 \longrightarrow$ quitenidine + formic acid. Hot alk. $KMnO_4 \longrightarrow$ pyridine-2 : 3 : 4-tricarboxylic acid, oxalic acid, CO_2, and NH_3. $CrO_3 \longrightarrow$ quininone + quininic acid. $PCl_5 \longrightarrow$ quinidine chloride. Gives thalleioquin reaction. 60% $H_2SO_4 \longrightarrow$ cupreidine + isocupreidine.

B_2,H_2SO_4: needles or prisms + $2H_2O$ from hot H_2O. Loses H_2O at 120°. Sol. 108 parts H_2O at 10°, 100 parts H_2O at 15°, 7 parts H_2O at 100°. Sol. 20 parts $CHCl_3$ at 15°. Sol. EtOH. Insol. Et_2O. $[\alpha]_D^{17}$ + 211·5° in EtOH. $[\alpha]_D^{15}$ + 218·2° in 80% EtOH, $[\alpha]_D^{15}$ + 179·5° in H_2O, $[\alpha]_D$ + 184·17° in $CHCl_3$.

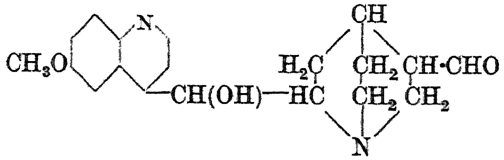
B,H_2SO_4: prisms + $4H_2O$. Sol. 8·7 parts H_2O at 10° with blue fluor.

B,HCl: prisms + H_2O. Sol. hot H_2O, EtOH. Prac. insol. Et_2O. $[\alpha]_D^{15}$ + 200·9° (+ 195·8°) in H_2O. Also needles + $2H_2O$, m.p. anhyd. 258–9° Sol. 75 parts H_2O at 15°. $[\alpha]_D$ + 212° in EtOH, $[\alpha]_D^{20}$ + 200° in H_2O.

B,2HCl : prisms + H_2O. Sol. EtOH. Spar. sol. $CHCl_3$. $[\alpha]_D^{15}$ + 250·3° in H_2O.
B,HBr : sol. 200 parts H_2O at 14°.
B,HI : prisms. Sol. 1270 parts H_2O at 15°. Prac. insol. EtOH.
B,2HI : golden prisms + $3H_2O$. Sol. 90 parts H_2O at 15°.
B,HNO₃ : prisms. Sol. 85 parts H_2O at 15°.
B,H₃PO₄ : prisms. Sol. 131 parts H_2O at 10°. Mod. sol. EtOH.
B₂,H₂CrO₄ : yellow plates + $6H_2O$.
B₂,H₂PtCl₆ : orange needles + $3H_2O$.
B,2HCl,CuCl₂ : orange plates. M.p. 208–9° decomp.
B₂,(COOH)₂ : cryst. + H_2O from H_2O. Sol. 151 parts H_2O at 15°. $[\alpha]_D^{15}$ + 186·8° in $CHCl_3$–EtOH.
Benzoate : cryst. + H_2O. Sol. EtOH.
Salicylate : sol. 1650 parts H_2O at 25°.
Acetyl : amorph. Sol. Et_2O. $[\alpha]_D^{15}$ + 128° in EtOH. *B,HAuCl₄* : yellow amorph. + $2H_2O$. *B,H₂PtCl₆* : yellow cryst. + $3H_2O$. Spar. sol. H_2O.
Salicyloyl : powder. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. *Salicylate* : needles from EtOH. M.p. 168°.
Methochloride : m.p. 236–7°.
α-Methiodide : needles + H_2O. M.p. 248° decomp. Sol. hot H_2O, EtOH.
Dimethiodide : yellow plates + $1\frac{1}{2}H_2O$. M.p. 156° decomp. More sol. H_2O and less sol. EtOH than monomethiodide.
α-Ethiodide : m.p. anhyd. 248° decomp. Sol. hot H_2O, EtOH.
Diethiodide : yellow prisms + $3H_2O$ from dil. EtOH. M.p. anhyd. 205° decomp. Sol. hot H_2O. Less sol. EtOH.

Hesse, *Ann.*, 1874, **174**, 337; *Ber.*, 1882, **15**, 3008.
Butler, Cretcher, *J. Am. Pharm. Assocn.*, 1933, **22**, 414.
Cohen, *J. Chem. Soc.*, 1933, 999.

Quininal

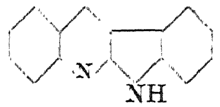


$C_{19}H_{22}O_3N_2$ MW, 326

Cryst. M.p. 160°. $[\alpha]_D^{19}$ − 30° in $CHCl_3$.
Phenylhydrazone : m.p. 145–7°.
p-*Bromophenylhydrazone* : m.p. 148–50°.
Acetyl: m.p. about 120° decomp. $[\alpha]_D^{22}$ − 63·5° in $CHCl_3$.
Benzoyl: m.p. 126°. $[\alpha]_D^{22\cdot4}$ + 79·4° in $CHCl_3$.
Chloropicrate : m.p. 130°.
Dichloropicrate : m.p. 126° decomp.

Seekles, *Rec. trav. chim.*, 1923, **42**, 99.

Quinindoline

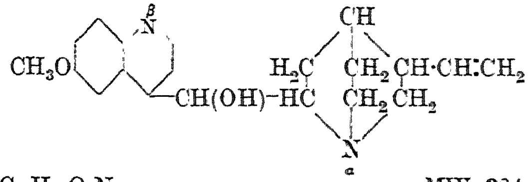


$C_{15}H_{10}N_2$ MW, 218

Yellow needles or leaflets from $PhNO_2$. M.p. 346° (342–3°). Sol. boiling $PhNO_2$, aniline. Prac. insol. hot C_6H_6, AcOEt, EtOH, $CHCl_3$, Et_2O. Sublimes. Sol. H_2SO_4 with violet fluor.
B,HCl : yellow needles. M.p. about 280° decomp.
B₂,H₂PtCl₆ : yellow cryst.
Acetyl : needles. M.p. 185°. Sol. C_6H_6. Spar. sol. MeOH.

Gabriel, Eschenbach, *Ber.*, 1897, **30**, 3020.
Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, **125**, 634.
Friedländer, Sander, *Ber.*, 1924, **57**, 652.

Quinine



$C_{20}H_{24}O_2N_2$ MW, 324

The most important of the cinchona alkaloids. Stereoisomeric with quinidine. Cryst. + $3H_2O$ from Et_2O below 10°, or needles + $3H_2O$ from EtOH. Loses $1H_2O$ above 15°, $2H_2O$ over H_2SO_4, and $3H_2O$ at 125°. The commercial product is usually a microcryst. powder + $2H_2O$. *Trihydrate* : m.p. 57° : sol. 1670 parts H_2O at 15°, 0·8 part EtOH, 1·1 parts $CHCl_3$, 1·9 parts Et_2O, 212 parts glycerol and 166 parts C_6H_6 at 25°. $[\alpha]_D^{15}$ − 145·2° in EtOH. *Anhydrous* : m.p. 177° (176°, 174·9°, 172·8°) : sol. 1960 parts H_2O at 15°, 760 parts H_2O at 100°, 1·1 parts EtOH, 22·6 parts Et_2O, 1·9 parts $CHCl_3$, 200 parts C_6H_6, 189 parts CCl_4 at 18–22°, 3450 parts 5% KOH and 1890 parts 10% NH_3 at 25°. Sol. dil. H_2SO_4 with blue fluor. $[\alpha]_D^{15}$ − 158·7° in Et_2O, − 169·3° in 97% EtOH. $[\alpha]_D^{17}$ − 167·5° in EtOH, − 117° in $CHCl_3$. Bitter taste. Gives thalleioquin reaction. Acid $KMnO_4$ in cold ⟶ quitenine + formic acid. Alk. $KMnO_4$ ⟶ pyridine-2 : 3 : 4-tricarboxylic acid. CrO_3 ⟶ quininone + quininic acid. PCl_5 ⟶ quinine chloride. Boiling AcOH ⟶ quinicine. Red. ⟶ hydroquinine. Br ⟶ quinine dibromide. HCl ⟶ apoquinine. HNO_3 ⟶ cinchomeronic acid. KOH fusion ⟶ 6-methoxylepidine + 6-methoxyquinoline. Forms add. comps. with C_6H_6 and other aromatic compounds. (*Note* : quinine is the Me ether of cupreine, but on demethylation apoquinine is

formed, which is also obtained when cupreine is treated with hydrogen halides.)

B_2,H_2SO_4: silky needles + $8H_2O$. Bitter taste. Loses $6H_2O$ on exposure to air giving dihydrate, m.p. 205°. Dehydrated completely at 100°. Sol. 740 parts H_2O at 13°, 30 parts H_2O at 100°, 65 parts EtOH at 15°, 24 parts glycerol at 15°, 1000 parts $CHCl_3$ at 15°. Readily sol. $CHCl_3$–EtOH (2 : 1). $[\alpha]_D^{17}$ − 157·4° in EtOH. $[\alpha]_D^{15}$ (anhyd.) − 235° in dil. H_2SO_4. Sol. dil. H_2SO_4 with strong blue fluor. Pptd. by potassium chromate, oxalate or picrate, sodium salicylate, tannic acid, Rochelle salt, etc.

B,H_2SO_4: prisms + $7H_2O$ from H_2O or EtOH. Loses $6H_2O$ over H_2SO_4 and $7H_2O$ at 100°. Softens at 60°, m.p. anhyd. 160°. Bitter taste. Shows strong fluor. in aq. sol. Sol. 11 parts H_2O, 45 parts EtOH at 13°, 18 parts glycerol, 920 parts $CHCl_3$, 1770 parts Et_2O at 25°. $[\alpha]_D^{15}$ − 164·5° in H_2O.

$B,2H_2SO_4$: prisms + $7H_2O$ from H_2O. Spar. sol. EtOH. Insol. Et_2O. Also prisms + $5H_2O$ from EtOH. $[\alpha]_D^{15}$ − 168·4° in H_2O.

B,HCl: needles + $2H_2O$. Loses H_2O at 100°. M.p. anhyd. 158–60°. Sol. 40 parts H_2O, 9 parts $CHCl_3$, 3 parts EtOH at 15°. Sol. 1 part boiling H_2O. $[\alpha]_D^{17}$ − 133·7° in H_2O, − 57·1° in $CHCl_3$. $[\alpha]_D^{15}$ − 145·5° in 97% EtOH.

B,2HCl: needles. Turns brown at 165–75°, m.p. 180–5°. Sol. 0·75 part H_2O, 5 parts EtOH, 7 parts $CHCl_3$ at 15°. Insol. Et_2O. $[\alpha]_D^{17-19}$ − 233° in H_2O.

B,HBr: needles + H_2O. Softens at 152°, melting finally at 200°. Sol. 55 parts H_2O, 7 parts EtOH, 10 parts $CHCl_3$ at 15°. Sol. 1 part boiling H_2O. Spar. sol. Et_2O. Hygroscopic.

B,2HBr: yellow prisms + $3H_2O$. M.p. 81–2°. Sol. 7 parts H_2O at 15°. Sol. EtOH. Insol. Et_2O.

B,HI: yellow needles. Sol. EtOH, Et_2O. Spar. sol. cold H_2O.

B,2HI: yellow prisms + $5H_2O$. M.p. about 100°. Sol. 20 parts H_2O at 15°.

B,HNO_3: cryst. + H_2O from H_2O. Sol. 70 parts H_2O at 25°.

B,H_2CO_3: needles + H_2O. Sol. EtOH.

B_2,H_3PO_4: needles + $8H_2O$ from H_2O. Sol. 784 parts H_2O at 10°.

B,H_3PO_2: sol. 60 parts H_2O at 15°.

B_2,H_2PtCl_6: orange, amorph. + $3H_2O$. Prac. insol. cold H_2O.

B,H_2PtCl_6: yellow cryst. + H_2O. Loses H_2O at 100°. Decomp. above 100°.

B_2,H_2CrO_4: yellow cryst. + $2H_2O$. Sol. 2400 parts H_2O at 15°, 160 parts H_2O at 100°. Sol. EtOH. Insol. Et_2O.

$B,2HCl,CuCl_2$: brick-red needles. M.p. 210° decomp.

Formate: B,H·COOH. Quinaform. Needles. M.p. 109° decomp. Sol. 19 parts H_2O at 16°, 8 parts H_2O at 32°. $[\alpha]_D^{20}$ − 144·2° in H_2O. B,2H·COOH: needles. Decomp. at 50°.

Acetate: B,CH_3COOH. Needles. M.p. 140°. Sol. 30 parts H_2O at 15°.

Chloroacetate: cryst. + $2\frac{1}{2}H_2O$. Sol. 64 parts H_2O at 21°. Sol. hot EtOH.

Dichloroacetate: needles + $2H_2O$. Sol. 41·4 parts H_2O at 22°.

Trichloroacetate: needles. M.p. 139–40°. Sol. H_2O, EtOH.

Oxalate: $B_2,(COOH)_2$. Prisms + $6H_2O$. Sol. 1030 parts H_2O at 10°.

d-*Tartrate*: $B_2,C_4H_6O_6$. Cryst. powder + $2H_2O$. Loses $1H_2O$ at 120° and second H_2O at 140°. Spar. sol. H_2O. $[\alpha]_D^{17}$ − 216·6° in dil. HCl. $B,C_4H_6O_6$: cryst. + H_2O. Loses H_2O at 160°. Less sol. H_2O than *l*-tartrate.

l-*Tartrate*: $B,C_4H_6O_6$. Cryst. + H_2O. Loses H_2O at 100°. More sol. H_2O than *d*-tartrate.

Citrate: $B,C_6H_8O_7$: needles from H_2O. M.p. 204° part decomp. $B_2,C_6H_8O_7$. Prisms or needles + $7H_2O$. Sol. 930 parts H_2O at 12°. $B_3,2C_6H_8O_7$: cryst.

l-*Lactate*: $B,C_3H_6O_3$. Needles + $\frac{1}{2}H_2O$ from H_2O.

dl-*Lactate*: $B,C_3H_6O_3$. Cryst. + H_2O.

Benzoate: $B,C_7H_6O_2$. Prisms. Sol. 373 parts H_2O at 100°.

Salicylate: $B,C_7H_6O_3$. Prisms from EtOH. Sol. 225 parts H_2O at 16°. Also cryst. + $2H_2O$ from H_2O. M.p. about 195°.

Acetylsalicylate: $B,C_9H_8O_4$. Xaxaquin. M.p. 157°. Insol. Et_2O.

Acetyl: cryst. from pet. ether. M.p. 116–17° (108°). Sol. EtOH, $CHCl_3$. Spar. sol. Et_2O. Almost tasteless. $[\alpha]_D^{15}$ − 54·3° in 97% EtOH, $[\alpha]_D^{22\cdot9}$ − 120·8° in dil. HCl. Sol. dil. acids.

Propionyl: prisms from Et_2O. M.p. 129°. Sol. $CHCl_3$. Mod. sol. EtOH, Et_2O. Prac. insol. H_2O.

Isovaleryl: amorph. Hygroscopic. Sol. EtOH, Et_2O, C_6H_6.

Benzoyl: prisms from Et_2O. M.p. 139° (138°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, pet. ether, CS_2. Insol. H_2O. Sol. min. acids. $[\alpha]_D^{17}$ + 121·6° in EtOH, $[\alpha]_D^{19\cdot2}$ + 119·9° in EtOH.

Salicyloyl: saloquinine. Cryst. from dil. EtOH or Et_2O. M.p. 140°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O. *Salicylate*: $B,C_7H_6O_3$. Rheumatin. Needles. M.p. 179°. Tasteless. Spar. sol. H_2O.

Anisoyl: needles from Et_2O. M.p. 87–8°. Sol. EtOH, $CHCl_3$, C_6H_6.

Cinnamoyl: needles from Et_2O. M.p. 111°. Sol. hot H_2O, EtOH, Et_2O, C_6H_6. Spar. sol. cold H_2O.

α-*Methochloride*: needles + H_2O. M.p. 181–2°. Sol. H_2O, EtOH.

α-*Methobromide*: needles + H_2O. M.p. 124–6°. Sol. EtOH. Mod. sol. cold H_2O.

α-*Methiodide*: needles + 1 or $2H_2O$ from H_2O. M.p. 233–6° decomp. Sol. EtOH. Spar. sol. cold H_2O. Prac. insol. Et_2O, $CHCl_3$.

Dimethioidide: yellow plates + $3H_2O$. M.p.

167–8° decomp. (158–62° decomp.). $[\alpha]_D^{18}$ − 151° in $2N.H_2SO_4$.

α-Ethochloride: needles + $3H_2O$. $[\alpha]_D$ − 122° in H_2O.

α-Ethobromide: cryst. + $2H_2O$. $[\alpha]_D$ − 117° in H_2O.

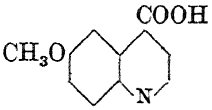
α-Ethiodide: cryst. + H_2O. M.p. anhyd. 210–11°. $[\alpha]_D$ − 105° in EtOH.

β-Ethiodide: needles + $3H_2O$. M.p. 93°.

Diethiodide: yellow cryst. + $3H_2O$ from dil. EtOH. M.p. 140°. Sol. hot H_2O, EtOH. Insol. Et_2O.

De Vrij, *Jahresber. Fortschr. Chem.*, 1864, 443.
Hesse, *Ann.*, 1890, **258**, 133.
Grimaux, Arnaud, *Bull. soc. chim.*, 1892, 7, 306.
Seekles, *Rec. trav. chim.*, 1923, **42**, 72.
Cohen, *J. Chem. Soc.*, 1933, 999.
Kindler, *Chem.-Ztg.*, 1932, **56**, 165.
Woodward, Doering, *J. Am. Chem. Soc.*, 1945, **67**, 860.

Quininic Acid (*6-Methoxycinchoninic acid, 6-hydroxyquinoline-4-carboxylic acid methyl ether*)



$C_{11}H_9O_3N$ MW, 203

Yellow prisms from dil. HCl. M.p. 285° decomp. Sol. acids with yellow col. Sol. alkalis. Spar. sol. EtOH with blue fluor. destroyed by H_2O or acids. Spar. sol. cold H_2O, Et_2O, C_6H_6. $k = 9 \times 10^{-6}$ at 25°. Alk. $KMnO_4 \longrightarrow$ pyridine-2 : 3 : 4-tricarboxylic acid. Conc. HCl $\longrightarrow$ 6-hydroxycinchoninic acid.

Me ester: $C_{12}H_{11}O_3N$. MW, 217. Prisms from EtOH or Et_2O. M.p. 85°.

Et ester: $C_{13}H_{13}O_3N$. MW, 231. M.p. 69°. Insol. H_2O. *B,HCl*: yellow needles from EtOH. M.p. 160° decomp. *B_2,H_2PtCl_6*: orange cryst. + $2H_2O$. M.p. 228° decomp.

Amide: $C_{11}H_{10}O_2N_2$. MW, 202. Needles from AcOEt. M.p. 210–12°. Sol. EtOH. Spar. sol. H_2O, Et_2O. Insol. ligroin, C_6H_6.

Chloride: $C_{11}H_8O_2NCl$. MW, 221·5. *B,HCl*: yellow powder. Decomp. at 186°.

Nitrile: 6 - methoxy - 4 - cyanoquinoline. $C_{11}H_8ON_2$. MW, 184. Yellow needles from C_6H_6. M.p. 157°. Readily sol. EtOH, $CHCl_3$, toluene. Sol. Et_2O, ligroin. Insol. H_2O.

Hydrazide: m.p. 151°.

B,HCl: yellow plates + $2H_2O$. M.p. 225°.

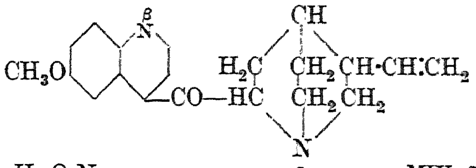
Methochloride: yellow needles. M.p. 215°. Sol. H_2O.

Methiodide: yellow plates or needles from EtOH or H_2O. M.p. 205° decomp. Sol. hot. H_2O, EtOH.

Ethobromide: yellow needles from EtOH. M.p. 210°.

Skraup, *Monatsh.*, 1881, **2**, 592.
Hirsch, *Monatsh.*, 1896, **17**, 327.
Koenigs, Schönewald, *Ber.*, 1902, **35**, 2986.
Kaufmann, Peyer, *Ber.*, 1912, **45**, 1805.
John, *Ber.*, 1930, **63**, 2657.
John, Andraschko, *J. prakt. Chem.*, 1930, **128**, 180.
Rabe, Huntenburg, Schultze, Volger, *Ber.*, 1931, **64**, 2492.
Thielepape, Fulde, *Ber.*, 1939, **72B**, 1432.
King, Work, *J. Chem. Soc.*, 1942, 401.
Elderfield *et al.*, *J. Org. Chem.*, 1946, **11**, 803.

Quininone (*6-Methoxycinchoninone*)



$C_{20}H_{22}O_2N_2$ MW, 322

Needles or leaflets. M.p. 108° (101–8° slow heat.). $[\alpha]_D^{23}$ + 73·8° in EtOH. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin.

B,HCl: hygroscopic cryst. M.p. 210–12°. $[\alpha]_D^{14}$ + 58·7° in EtOH.

Oxime: m.p. 113° (not sharp).

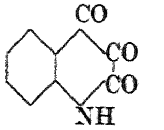
Picrate: cryst. from EtOH. M.p. 232–3°.

Picrolonate: yellow needles from EtOH. M.p. 197–8°.

α-Methiodide: cryst. from MeOH. M.p. 213–14°.

Rabe, Kuliga, *Ann.*, 1909, **364**, 346, 349.
Woodward, Wendler, Brutschy, *J. Am. Chem. Soc.*, 1945, **67**, 1425.

Quinisatin (2 : 3 : *4-Triketotetrahydroquinoline*)



$C_9H_5O_3N$ MW, 175

Yellow prisms + H_2O from H_2O. Loses H_2O at 120–5° turning red. M.p. 255–60°. Mod. sol. cold, sol. hot H_2O. Anhyd. sol. EtOH. Sol. NaOH aq. with yellow col. $NH_2OH \longrightarrow$ 3-oxime.

N-Me: reddish-yellow cryst. M.p. 120–2°. Sol. alkalis with yellow col. *Oxime*: red needles from AcOH. Decomp. at 188°. Sol. alkalis with green col.

3-Oxime: orange-yellow prisms from EtOH. M.p. 208° decomp. Sol. AcOH. Spar. sol. H_2O, EtOH, Et_2O, $CHCl_3$, C_6H_6. Sol. alkalis with reddish-brown col. Sol. H_2SO_4 with red col.

Sn + HCl ⟶ 3-amino-2 : 4-dihydroxyquinoline.

Baeyer, Homolka, *Ber.*, 1883, **16**, 2219; 1884, **17**, 985.
Friedländer, Müller, *Ber.*, 1887, **20**, 2015.
See also Kalb, *Ber.*, 1911, **44**, 1460.

Quinisoamyline.
See under Cupreine.

Quinisopropyline.
See under Cupreine.

Quinitol (*Hexahydrohydroquinone, cyclohexandiol*-1 : 4, 1 : 4-*dihydroxycyclohexane*)

$$HO{\cdot}CH\!<\!\begin{matrix}CH_2—CH_2\\CH_2—CH_2\end{matrix}\!>\!CH{\cdot}OH$$

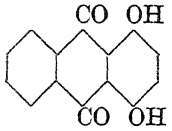
$C_6H_{12}O_2$ MW, 116

Cis :
Prisms from Me_2CO. M.p. 112·4–112·8°. Sol. H_2O, EtOH, Me_2CO. Spar. sol. Et_2O, $CHCl_3$. Sublimes in vacuo. Stable to $KMnO_4$.
Diacetyl : cryst. from dil. EtOH. M.p. 41·25°. B.p. 251°, 145–7°/25 mm., 130°/15 mm. n_D^{25} (supercooled) 1·4508.
Dipropionyl : m.p. 39·5–40°.
Dibenzoyl : m.p. 116–17°.
Dicinnamoyl : m.p. 122°.
Me ether : $C_7H_{14}O_2$. MW, 130. B.p. 102–3°/15 mm. n_D^{19} 1·4671. D^{19} 1·023.
Di-Me ether : $C_8H_{16}O_2$. MW, 144. B.p. 67·5–68°/14 mm. n_D^{18} 1·4440. D^{18} 0·9526.
Mono-p-*toluenesulphonyl* : needles from C_6H_6–pet. ether. M.p. 94–5°.
Di-p-*toluenesulphonyl* : needles from MeOH. M.p. 98–9°.

Trans :
Plates from Me_2CO. M.p. 143°. Sol. H_2O, EtOH. Spar. sol. cold Me_2CO. Very spar. sol. Et_2O.
Diacetyl : cryst. from EtOH. M.p. 102–3°. B.p. 245–50°/710 mm., 145–7°/25 mm.
Dipropionyl : m.p. 75·5–76°.
Dibenzoyl : m.p. 151°.
Dicinnamoyl : m.p. 189°.
Me ether : b.p. 102·5–103°/15 mm. n_D^{20} 1·4649. D^{19} 1·021.
Di-Me ether : b.p. 68–9°/15 mm. n_D^{18} 1·4430.
Mono-p-*toluenesulphonyl* : needles from C_6H_6–pet. ether. M.p. 111°.
Di-p-*toluenesulphonyl* : needles from C_6H_6. M.p. 159°.

Sabatier, Mailhe, *Ann. chim. phys.*, 1909, **16**, 90.
Leroux, *Ann. chim. phys.*, 1910, **21**, 542.
Baeyer, *Ann.*, 1894, **278**, 92.
Willstätter, Lessing, *Ber.*, 1901, **34**, 507.
Uspenski, Turin, *Chem. Zentr.*, 1923, III, 754.
Palfray, Sabetay, *Bull. soc. chim.*, 1928, **43**, 898.
Palfray, Rothstein, *Bull. soc. chim.*, 1929, **45**, 855.
Rothstein, *Ann. chim.*, 1930, **14**, 486.
Olberg, Pines, Ipatieff, *J. Am. Chem. Soc.*, 1944, **66**, 1096.
Perrine, White, *J. Am. Chem. Soc.*, 1947, **69**, 1542.
Owen, Robins, *J. Chem. Soc.*, 1949, 320.

Quinizarin (1 : 4-*Dihydroxyanthraquinone*)

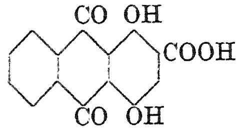


$C_{14}H_8O_4$ MW, 240

Red. cryst. from AcOH, m.p. 200–2°; red cryst. from toluene, m.p. 194°. Sol. 12–13 parts boiling AcOH. Sol. alkalis with violet-blue col. Sol. conc. H_2SO_4 with violet-red col. and greenish-yellow fluor. PbO_2 in AcOH ⟶ anthra-1 : 4 : 9 : 10-diquinone. HNO_3 ⟶ 2-nitro deriv. $K_3Fe(CN)_6$ ⟶ phthalic acid. Hydrosulphite ⟶ leucoquinizarin. Intermediate for dyestuffs.
Me ether : $C_{15}H_{10}O_4$. MW, 254. Yellow needles from MeOH. M.p. 189° (167–8°). Sol. alkalis.
Di-Me ether : $C_{16}H_{12}O_4$. MW, 268. Cryst. from C_6H_6. M.p. 170–1° (143°).
Et ether : $C_{16}H_{12}O_4$. MW, 268. Red needles from EtOH. M.p. 150–1°. Mod. sol. alkalis with reddish-violet col.
Di-Et ether : $C_{18}H_{16}O_4$. MW, 296. Yellow needles. M.p. 176–7°.
Diphenyl ether : 1 : 4-diphenoxyanthraquinone. $C_{26}H_{16}O_4$. MW, 392. Yellow needles from EtOH. M.p. 165°. Sol. boiling EtOH, C_6H_6, AcOH. Spar. sol. boiling MeOH. Insol. ligroin. Sol. conc. H_2SO_4 with bluish-violet col.
Acetyl : orange-yellow needles from C_6H_6. M.p. 186°.
Diacetyl : orange-yellow cryst. from AcOH. M.p. 207–8° (200°). Exists in polymorphic forms.
1-*Acetyl*-4-*benzoyl* : m.p. 195–6°.

Baeyer, Caro, *Ber.*, 1875, **8**, 152.
Liebermann, *Ann.*, 1882, **212**, 10.
Bayer, D.R.P., 229,316, (*Chem. Zentr.*, 1911, I, 181); D.R.P., 255,031, (*Chem. Zentr.*, 1913, I, 354).
M.L.B., D.R.P., 242,379, (*Chem. Zentr.*, 1912, I, 301).
Eckert, Steiner, *Monatsh.*, 1914, **35**, 1145.
Green, *J. Chem. Soc.*, 1926, 1428.
Bigelow, Reynolds, *Organic Syntheses*, Collective Vol. I, 464.
National Aniline and Chemical Co., U.S.P., 1,886,237, (*Chem. Abstracts*, 1933, **27**, 1366).
British Celanese, B.P. 346,355, (*Chem. Abstracts*, 1932, **26**, 1948).
United Alkali, B.P. 245,584, (*Chem. Abstracts*, 1927, **21**, 249).
du Pont, U.S.P., 2,003,859, (*Chem. Abstracts*, 1935, **29**, 4776).

Quinizarin-2-carboxylic Acid (1 : 4-*Dihydroxyanthraquinone*-2-*carboxylic acid*)



$C_{15}H_8O_6$ MW, 284

Red or yellowish-brown needles from $PhNO_2$. M.p. 249–50° (244–6°). Sol. Me_2CO, warm AcOH, toluene. Spar. sol. EtOH, Et_2O, C_6H_6. Sol. alkalis. Heat ⟶ quinizarin.

Ullmann, Schmidt, *Ber.*, 1919, **52**, 2111.
Bayer, D.R.P., 273,341, (*Chem. Zentr.*, 1914, I, 1719).

Quinizarin-5-carboxylic Acid (5 : 8-*Dihydroxyanthraquinone*-1-*carboxylic acid*, 1 : 4-*dihydroxyanthraquinone*-5-*carboxylic acid*).

Red needles from *p*-xylene. Decomp. at 250°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O.

Kögl, Deijs, *Ann.*, 1935, **515**, 31.

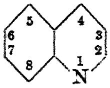
Quinizarin-6-carboxylic Acid (5 : 8-*Dihydroxyanthraquinone*-2-*carboxylic acid*, 1 : 4-*dihydroxyanthraquinone*-6-*carboxylic acid*).

Orange brown needles from AcOH. Sol. Na_2CO_3 ⟶ violet red col. Sol. NaOH ⟶ blue col.

Bayer, D.R.P. 84,505.

Quinol.
See Hydroquinone.

Quinoline (2 : 3-*Benzpyridine*)



C_9H_7N MW, 129

Occurs in coal-tar and in "stupp" fat. F.p. − 20° (− 15°). B.p. 238·05°/760 mm., 236·9°/749·2 mm., 114°/17 mm. Sol. hot H_2O, dil. acids, EtOH, Et_2O, Me_2CO, CS_2. Mod. sol. cold H_2O. Hygroscopic. Volatile in steam. D_0^0 1·1081, D_0^{20} 1·0947, D_4^{20} 1·0929, D_4^{25} 1·0900 (1·08979), D_4^{50} 1·0699. n_D^{11} 1·6305, n_D^{20} 1·6268, $n_D^{24·9}$ 1·6245, n_D^{29} 1·6218. Heat of comb. C_v 1122·3 Cal. $KMnO_4$ ⟶ quinolinic acid, oxalic acid, NH_3, and CO_2. $HNO_3 + H_2SO_4$ ⟶ 5- + 8-nitroquinolines ⟶ 5 : 7- + 6 : 8-dinitroquinolines. H_2SO_4 at 220° ⟶ quinoline-8-sulphonic acid + a small quantity of quinoline-5-sulphonic acid. H_2SO_4 at 300° ⟶ quinoline-6-sulphonic acid. Sn + HCl or Na + EtOH ⟶ 1 : 2 : 3 : 4-tetrahydroquinoline. HI(+ P) ⟶ hexa- and decahydroquinolines. H(+ Ni) ⟶ 1 : 2 : 3 : 4-tetrahydroquinoline ⟶ decahydroquinoline.

B,HCl : deliquescent prisms + $\frac{1}{2}H_2O$. M.p. 94°, anhyd. 134·5°. Sol. EtOH, $CHCl_3$. Spar. sol. Et_2O–C_6H_6.

B_2,3HCl : m.p. 82°.

B,2HCl : m.p. 46·7° (48·5–57°).

B_2,HBr : cryst. + $2H_2O$. M.p. 41°.

B,HI : yellow needles from EtOH. M.p. 135°.

B,H_2SO_4 : cryst. from EtOH or AcOH. M.p. 163·5–164·5°.

B,HNO_3 : needles from EtOH.

B,HSCN : plates. M.p. 140°. Spar. sol. H_2O, EtOH.

B_2,$H_2Cr_2O_7$: yellow needles from H_2O. M.p 167° (165–7°). Sol. 282 parts H_2O at 10·5°

B,HCl,$CuCl_2$: brown needles. Decomp. at 185°.

B_2,2HCl,$AuCl_3$: cryst. ppt. M.p. 180°. Decomp. at about 260°.

B,$AuCl_3$: yellow cryst. from EtOH.

B,$HAuCl_4$: yellow needles. M.p. 235–8°. Decomp. at about 260°. Spar. sol. cold H_2O.

B_2,H_2PtCl_6 : orange-yellow needles + $2H_2O$ from dil. HCl. M.p. 227·5° (225°, 226°, 218°).

B_2,H_2PtBr_6 : red prisms or needles. M.p. 254–5°.

Dichloroacetate : cryst. M.p. 63–4°. Sol. H_2O.

Trichloroacetate : cryst. from EtOH. M.p. 100°. Sol. H_2O.

B,$(COOH)_2$: needles from EtOH. M.p. about 105°.

d-*Tartrate* : cryst. + $5H_2O$. M.p. 131°. $[\alpha]_D^{20}$ + 13·2° in H_2O ($[\alpha]_D^{15}$ + 13·5° in H_2O).

Acid d-*tartrate* : $[\alpha]_D^{15}$ + 14·9° in H_2O.

Acid phthalate : cryst. M.p. 98–9°. Sol. EtOH.

Picrate : yellow needles from C_6H_6. M.p. 203°.

Styphnate : m.p. 207–8°.

Methochloride : cryst. + H_2O from EtOH. M.p. 126°.

Methobromide : needles. M.p. 70°. Sol. H_2O, EtOH, Et_2O, $CHCl_3$. Spar. sol. Me_2CO. Prac. insol. C_6H_6, pet. ether.

Methiodide : orange-red cryst. from EtOH, m.p. 133° (144·5°); yellow cryst. + H_2O from EtOH, m.p. 72°; yellow cryst. + C_6H_6 from C_6H_6, m.p. about 133°.

Methopicrate : yellow needles from H_2O. M.p. 164–5° (169·5°).

Ethochloride : plates + H_2O. M.p. 92·5°, anhyd. 122°.

Ethobromide : plates + H_2O from H_2O or EtOH. M.p. 80°. Loses H_2O at 100°. Sol. $CHCl_3$. Insol. Et_2O.

Ethiodide : yellow prisms from EtOH. M.p. 158° (156–7°). Sol. H_2O, EtOH, $CHCl_3$. Insol. Et_2O.

Propyl chloride : plates + H_2O from H_2O, m.p. 95°. Loses H_2O at 130–5° and melts at 135°. The anhyd. salt is hydroscopic. Cryst. from $CHCl_3$ in prisms + $CHCl_3$, m.p. 79°.

Propyl bromide : hygroscopic cryst. from EtOH, m.p. 148°; plates + $2H_2O$ from H_2O or EtOH, m.p. 66°.

Propyl iodide : yellow cryst. from H_2O or

EtOH, m.p. 145°; yellow prisms + $CHCl_3$ from $CHCl_3$.

Isopropyl iodide: needles from Me_2CO. M.p. 136°.

Butyl iodide: yellow cryst. from EtOH or prisms from Me_2CO. M.p. 174°.

Isobutyl iodide: yellow cryst. from EtOH or prisms from Me_2CO. M.p. 161°.

Isoamyl bromide: yellow needles + H_2O from EtOH. M.p. 87°, anhyd. 140°.

Isoamyl iodide: yellowish-green cryst. M.p. 184–5°.

Hexadecyl iodide: m.p. 101°.

Allyl iodide: cryst. from H_2O or EtOH. M.p. 177·5°.

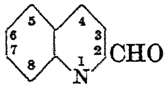
Benzyl chloride: plates + $3H_2O$ from H_2O. M.p. 65°, anhyd. 170°. Sol. H_2O, EtOH. Insol. Et_2O. $KMnO_4 \longrightarrow$ 2-benzylaminobenzoic acid.

Benzyl iodide: cryst. M.p. 135°.

N-*Oxide*: needles + $2H_2O$. M.p. 62°. *Picrate*: m.p. 143°.

Skraup, *Monatsh.*, 1880, **1**, 316; 1881, **2**, 141.
Walter, *J. prakt. Chem.*, 1894, **49**, 549.
Kneuppel, *Ber.*, 1896, **29**, 704.
Hantzsch, *Ber.*, 1909, **42**, 80.
Barnett, *Chem. News*, 1920, **121**, 205.
König, *Ber.*, 1923, **56**, 1853.
Cohn, Gustavson, *J. Am. Chem. Soc.*, 1928, **50**, 2709.
Cohn, *J. Am. Chem. Soc.*, 1930, **52**, 3685.
Clarke, Davis, *Organic Syntheses*, Collective Vol. I, 466.
Kirkhgof, Fedotov, *Chem. Abstracts*, 1933, **27**, 5331.
Darzens, Delaby, Hiron, *Bull. soc. chim.*, 1930, **47**, 227.
Kirchhof, Sassossow, *Chem. Zentr.*, 1935, I, 2371.
Mikhaĭlov, *Chem. Abstracts*, 1934, **28**, 3736.
Dehn, Cope, *J. Am. Chem. Soc.*, 1926, **48**, 2634.
Bert, *Compt. rend.*, 1942, **215**, 415.
Mikhaĭlov, *Chem. Abstracts*, 1940, **34**, 5847.

Quinoline-2-aldehyde (2-*Aldehydoquinoline*)



$C_{10}H_7ON$ MW, 157

Prisms or plates from pet. ether, m.p. 71° (70–1°, 67–9°): needles + ?H_2O from H_2O, m.p. 51°. Sol. EtOH, C_6H_6. Spar. sol. H_2O, pet ether. Reduces $NH_3.AgNO_3$.

Oxime: needles. M.p. 189° (184°).

Phenylhydrazone: yellow plates from EtOH. M.p. 204° (195–8°). Spar. sol. cold EtOH. Prac. insol. H_2O, Et_2O.

p-*Nitrophenylhydrazone*: yellow cryst. Sublimes at 225°. M.p. 250°.

Dinitrophenylhydrazone: yellow cryst. M.p. 251–3°.

Semicarbazone: m.p. 232–4° decomp.

Einhorn, *Ber.*, 1886, **19**, 908.
v. Miller, Spady, *Ber.*, 1885, **18**, 3404; 1886, **19**, 132.
Hammick, *J. Chem. Soc.*, 1926, 1303.
Monti, *Chem. Abstracts*, 1934, **28**, 4733.
Cooper, Cohen, *J. Chem. Soc.*, 1932, 723.
Pfitzinger, *J. prakt. Chem.*, 1902, **66**, 264.
Kaufmann, Valette, *Ber.*, 1913, **46**, 57.
Kwartler, Lindwall, *J. Am. Chem. Soc.*, 1937, **59**, 524.
Kaplan, *J. Am. Chem. Soc.*, 1941, **63**, 2654.

Quinoline-3-aldehyde (3-*Aldehydoquinoline*).

M.p. 70°.

Cook, Heilbron, Steger, *J. Chem. Soc.*, 1943, 413.

Quinoline-4-aldehyde (4-*Aldehydoquinoline*).

Plates + $1H_2O$, m.p. 84–84·5°. Sol. H_2O, EtOH, Et_2O, toluene. Anhyd. needles from toluene–pet. ether, m.p. 51–3°. B.p. 122–3°/4 mm., 98–108°/0·1 mm. Sol. Et_2O, toluene, xylene. Reduces Tollen's.

B,HCl: colourless prisms from EtOH–AcOH. M.p. 206°.

Oxime: needles from MeOH. M.p. 181–2°.

Phenylhydrazone: yellow cryst. M.p. 175–6°.

p-*Nitrophenylhydrazone*: yellow prisms from EtOH. M.p. 261–2°.

Semicarbazone: m.p. 244–5° decomp.

Picrate: from EtOH. Sinters at 170°, m.p. 179°.

Kwartler, Lindwall, *J. Am. Chem. Soc.*, 1937, **59**, 524.
Clemo, Hoggarth, *J. Chem. Soc.*, 1939, 1241.
Kaplan, *J. Am. Chem. Soc.*, 1941, **63**, 2654.
Macdonald, *J. Am. Chem. Soc.*, 1947, **69**, 1219.

Quinoline-5-aldehyde (5-*Aldehydoquinoline*).

Needles from H_2O. M.p. 95·5–96·5°.

B,HCl: m.p. 218–19°.

Anil: m.p. 70–71°.

Oxime: m.p. 188–90°.

Hydrazone: m.p. 110–11·5°.

Phenylhydrazone: m.p. 188–9°.

Semicarbazone: m.p. 212–15°.

Cook, Heilbron, Steger, *J. Chem. Soc.*, 1943, 413.
Rodionov, Berkengeim, *Chem. Abstracts*, 1945, **39**, 4076.

Quinoline-6-aldehyde (*6-Aldehydoquinoline*).

Needles + H_2O from H_2O. M.p. 55°, anhyd. 75–6°.

B_2,H_2PtCl_6: reddish-yellow needles. M.p. 244°.

Anil: needles from dil. EtOH. M.p. 99°.

Oxime: yellow needles from EtOH. M.p. 191°.

Phenylhydrazone: reddish-yellow needles + H_2O from EtOH. M.p. 185°.

Semicarbazone: yellow needles from EtOH. M.p. 239°.

Azine: yellow needles from EtOH. M.p. 261°. Spar. sol. EtOH.

Methiodide: yellow cryst. from EtOH. M.p. 218°. Sol. H_2O.

Howitz, Philipp, *Ann.*, 1913, **396**, 28.
Cook, Heilbron, Steger, *J. Chem. Soc.*, 1943, 413.
Rodionov, Berkengeim, *Chem. Abstracts*, 1945, **39**, 4076.

Quinoline-7-aldehyde (*7-Aldehydoquinoline*).

M.p. 85–6°.

B,HCl: m.p. 187–9°.

Anil: m.p. 72–4°.

Oxime: m.p. 185–7°.

Hydrazone: m.p. 123–4°.

Phenylhydrazone: m.p. 170–1°.

Semicarbazone: m.p. 234°.

Rodionov, Berkengeim, *Chem. Abstracts*, 1945, **39**, 4076.

Quinoline-8-aldehyde (*8-Aldehydoquinoline*).

Needles from dil. EtOH. M.p. 94–5°. Sol. hot H_2O, EtOH, Et_2O. Volatile in steam.

B,HCl: cryst. from EtOH. M.p. 213°. Sol. H_2O, EtOH.

B,HI: red cryst. from H_2O. M.p. 228° decomp.

B_2,H_2PtCl_6: reddish-yellow cryst. from alc. HCl. M.p. 250° decomp.

Anil: yellow cryst. from EtOH. M.p. 82°.

Oxime: plates + $\frac{1}{2}H_2O$ from dil. EtOH. M.p. 115°.

Phenylhydrazone: yellow needles from EtOH. M.p. 176°.

Semicarbazone: needles from C_6H_6. M.p. 238–9°.

Azine: yellow needles from C_6H_6. M.p. 248–9°. Spar. sol. C_6H_6.

Howitz, *Ber.*, 1902, **35**, 1274.
Howitz, Schwenk, *Ber.*, 1905, **38**, 1289.
Howitz, Köpke, *Ann.*, 1913, **396**, 39.
Cook, Heilbron, Steger, *J. Chem. Soc.*, 1943, 413.
Rodionov, Berkengeim, *Chem. Abstracts*, 1945, **39**, 4076.

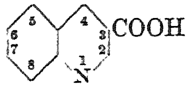
2-Quinoline-carbinol.

See 2-Hydroxymethyl-quinoline.

Quinoline-2-carboxylic Acid.

See Quinaldinic Acid.

Quinoline-3-carboxylic Acid



$C_{10}H_7O_2N$ MW, 173

Plates from dil. EtOH. M.p. 275° part. decomp. (271–2°). Sol. hot H_2O, EtOH. Spar. sol. cold H_2O. Alk. $KMnO_4$ ⟶ pyridine-2 : 3 : 5-tricarboxylic acid. Heat with lime ⟶ quinoline.

Me ester: $C_{11}H_9O_2N$. MW, 187. M.p. 76°. *Picrate*: m.p. 187–8°.

Et ester: $C_{12}H_{11}O_2N$. MW, 201. M.p. 69–69·5°.

Nitrile: 3-cyanoquinoline. $C_{10}H_6N_2$. MW, 154. M.p. 108°.

Amide: $C_{10}H_8ON_2$. MW, 172. Needles from H_2O. M.p. 198–9° (195°). Mod. sol. EtOH. *Picrate*: needles. M.p. 217–18°. Spar. sol. cold EtOH.

B_2,H_2PtCl_6: orange-yellow plates or needles. Sol. hot H_2O.

Hydrazide: m.p. 190°.

Doebner, v. Miller, *Ber.*, 1885, **18**, 1644.
Riedel, *Ber.*, 1883, **16**, 1613.
Graebe, Caro, *Ber.*, 1880, **13**, 101.
Mills, Watson, *J. Chem. Soc.*, 1910, **97**, 745.
Koller, Ruppersberg, Strang, *Monatsh.*, 1929, **52**, 66.

Quinoline-4-carboxylic Acid.

See Cinchoninic Acid.

Quinoline-5-carboxylic Acid.

Cryst. from hot AcOH. M.p. 342°. Spar. sol. EtOH, H_2O. Insol. Et_2O, C_6H_6, CS_2. Sublimes. Sol. dil. acids and alkalis. Heat with lime ⟶ quinoline.

Et ester: m.p. 10°. B.p. 190–2°/15 mm.

Nitrile: 5-cyanoquinoline. $C_{10}H_6N_2$. MW, 154. Needles from ligroin, m.p. 89° (87–8°): needles + $1\frac{1}{2}H_2O$ from dil. EtOH, m.p. 70°. Does not boil below 360°. Sol. EtOH, C_6H_6, CS_2. Spar. sol. H_2O, ligroin. *Hydrazide*: m.p. 169°. *Picrate*: m.p. 241°. Insol. H_2O.

B,HCl: needles or prisms + H_2O. Sol. EtOH.

B_2,H_2PtCl_6: yellow plates or needles. Spar. sol. H_2O.

Bedall, Fischer, *Ber.*, 1881, **14**, 2574; 1882, **15**, 683.
Jakubowski, *Ber.*, 1910, **43**, 3026.
Schlosser, Skraup, *Monatsh.*, 1881, **2**, 519.
Lellmann, Alt, *Ann.*, 1887, **237**, 318.
Bradford, Elliott, Rowe, *J. Chem. Soc.*, 1947, 437.
See also Skraup, Brunner, *Monatsh.*, 1886, **7**, 153, 519.

Quinoline-6-carboxylic Acid.

Prisms, plates or needles. M.p. 291–2° (290–1°). Sol. warm EtOH. Spar. sol. H_2O. Sublimes. Sol. dil. acids and alkalis.

Me ester: m.p. 90°.

Et ester: $C_{12}H_{11}O_2N$. MW, 201. Needles from dil. EtOH. M.p. 50°. *B,HCl*: needles from EtOH. M.p. 210° decomp. Sol. H_2O.

Amide: $C_{10}H_8ON_2$. MW, 172. Yellow plates from C_6H_6–EtOH. M.p. 174°. *Ethiodide*: red or greenish-yellow cryst. from dil. EtOH. M.p. 229° part. decomp.

Nitrile: cryst. from C_6H_6–ligroin. M.p. 131° (135°). Sublimes. Sol. dil. HCl with red col.

Hydrazide: m.p. 188°.

B,HCl: needles + H_2O. Hyd. by H_2O.

B_2,H_2PtCl_6: reddish-yellow plates.

Schlosser, Skraup, *Monatsh.*, 1881, **2**, 526.
v. Georgievics, *Monatsh.*, 1891, **12**, 306.
Biedermann, *Ber.*, 1889, **22**, 2762.
Einhorn, Feibelmann, *Ber.*, 1909, **42**, 4854.
Howitz, Philipp, *Ann.*, 1913, **396**, 29.
I.G., F.P., 727,528, (*Chem. Abstracts*, 1932, **26**, 5104).
Alamela, Dey, *Chem. Abstracts*, 1949, **43**, 1421.

Quinoline-7-carboxylic Acid.

Needles from H_2O or EtOH. M.p. 248–9° (248·5–250°, 247°). Sol. EtOH. Spar. sol. hot H_2O. Prac. insol. cold H_2O, C_6H_6. Insol. Et_2O. Sublimes. Heat with lime ⟶ quinoline.

Nitrile: pale brown cryst. M.p. 104°.

B,HCl: prisms + H_2O from HCl.Aq.

B_2,H_2PtCl_6: orange prisms or needles.

Skraup, Brunner, *Monatsh.*, 1886, **7**, 142.
Fischer, van Loo, *Ber.*, 1886, **19**, 2473.

Quinoline-8-carboxylic Acid.

Needles from H_2O. M.p. 187° (186–187·5°). Mod. sol. hot H_2O, EtOH. Sol. dil. acids and alkalis. Sublimes. $KMnO_4$ ⟶ quinolinic acid.

Et ester: m.p. 45°. B.p. 194–7°/13 mm., 145–55°/0·5 mm.

Nitrile: 8-cyanoquinoline. Needles from 50% EtOH. M.p. 84°. *B_2,H_2PtCl_6*: orange-yellow needles from H_2O.

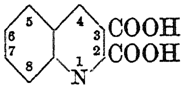
Hydrazide: m.p. 99°.

B,HCl: prisms from dil. HCl. Sol. H_2O. Spar. sol. dil. EtOH.

B_2,H_2PtCl_6: orange-yellow needles or red granular cryst. from dil. HCl. Spar. sol. H_2O.

Schlosser, Skraup, *Monatsh.*, 1881, **2**, 530.
Howitz, *Ber.*, 1902, **35**, 1275.
Bedall, Fischer, *Ber.*, 1882, **15**, 683.
Lellmann, Reusch, *Ber.*, 1889, **22**, 1391.
Chakravarti, Granapati, *Chem. Abstracts*, 1935, **29**, 1090.
Alamela, Dey, *Chem. Abstracts*, 1949, **43**, 1421.

Quinoline-2 : 3-dicarboxylic Acid (*Acridinic acid*)



$C_{11}H_7O_4N$ MW, 217

Needles + 2 or $3H_2O$ from hot H_2O or EtOH. Starts to lose CO_2 at 105° leaving residue, m.p. 274°. Sol. EtOH. Spar sol. cold H_2O, Et_2O. Decomp. at 120° ⟶ quinoline-3-carboxylic acid. Does not form salts with acids. Heat with lime ⟶ quinoline. $FeSO_4$ ⟶ reddish-yellow col.

Me ester: $C_{12}H_9O_4N$. MW, 231. M.p. 174–6°.

Di-Me ester: $C_{13}H_{11}O_4N$. MW, 245. Prisms from MeOH or C_6H_6. M.p. 107–8°.

Et ester: $C_{13}H_{11}O_4N$. MW, 245. M.p. 170–2°.

Di-Et ester: $C_{15}H_{15}O_4N$. MW, 273. Prisms from dil. EtOH. M.p. 55–6°.

Anhydride: $C_{11}H_5O_3N$. MW, 199. M.p. 225° (223°).

Amide: $C_{11}H_8O_3N_2$. MW, 216. Cryst. for H_2O or EtOH. M.p. 174–6° decomp.

Diamide: $C_{11}H_9O_2N_3$. MW, 215. Sinters at 250–70° ⟶ imide.

Anilide: m.p. 187° decomp.

Dianilide: sinters at 245°.

Imide: $C_{11}H_6O_2N_2$. MW, 198. M.p. 316° (314–15°).

Phenylimide: m.p. 319–20°.

Graebe, Caro, *Ber.*, 1880, **13**, 100.
Konopnicki, Sucharda, *Chem. Abstracts*, 1928, **22**, 785.
Koller, Strang, *Monatsh.*, 1928, **50**, 48.
Hozer, v. Niementowski, *J. prakt. Chem.*, 1927, **116**, 43.

Quinoline-2 : 4-dicarboxylic Acid.

Needles from H_2O. M.p. 246° decomp. Spar. sol. cold H_2O, EtOH, Et_2O. Insol. $CHCl_3$, C_6H_6, pet. ether. Heat at 240° ⟶ cinchoninic acid. Heat with lime ⟶ quinoline.

2-*Nitrile*: 2-cyanocinchoninic acid. $C_{11}H_6O_2N_2$. MW, 198. Needles. M.p. 226°.

Pfitzinger, *J. prakt. Chem.*, 1897, **56**, 308; 1902, **66**, 264.
Doebner, Peters, *Ber.*, 1889, **22**, 3009.

Quinoline-2 : 6-dicarboxylic Acid.

Cryst. M.p. 275–80° decomp.

v. Miller, *Ber.*, 1890, **23**, 2261.

Quinoline-5 : 6-dicarboxylic Acid.

Plates + H_2O from dil. HCl. M.p. 238–41°. Spar. sol. H_2O, EtOH, Et_2O.

Di-Me ester: m.p. 120–1°.

B,HCl: prisms for conc. HCl. Hyd. by H_2O.

B_2,H_2PtCl_6: brownish-yellow needles. Decomp. above 240°.

Hepner, *Monatsh.*, 1906, **27**, 1062.

Quinoline-5 : 8-dicarboxylic Acid.

Needles + $2H_2O$ from dil. HCl. M.p. 268–70°. Prac. insol. cold H_2O. At 270–80° ⟶ quinoline-5- and -8-carboxylic acids.

B,HCl: cryst. + $1\frac{1}{2}H_2O$. Hyd. by H_2O.

B_2,H_2PtCl_6: yellowish-red cryst. Decomp. by H_2O or EtOH.

Skraup, Brunner, *Monatsh.*, 1886, **7**, 149.

Quinoline-6 : 7-dicarboxylic Acid.

M.p. 240–50°.

Me ester: m.p. 104–5°.

Lindner, Sellner, Hofmann, Hager, *Monatsh.*, 1939, **72**, 335.

Quinoline-7 : 8-dicarboxylic Acid.

Prisms or needles + H_2O from H_2O. M.p. 206–7° decomp. Spar. sol. hot EtOH, cold H_2O. Part. decomp. on steam dist.

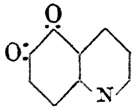
B,HCl: prisms. M.p. 212° decomp. Hyd. by H_2O.

Haid, *Monatsh.*, 1906, **27**, 333.

Quinoline-hydroquinone.

See 5 : 8-Dihydroxyquinoline.

Quinoline-5 : 6-quinone



$C_9H_5O_2N$ MW, 159

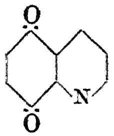
Prisms. Does not melt below 350°. Sol. AcOH and min. acids. Prac. insol. H_2O and most org. solvents.

5-*Oxime*: 5-nitroso-6-hydroxyquinoline. Golden-yellow needles from EtOH or AcOH. Chars above 180° without melting. Sol. dil. acids and alkalis. Spar. sol. Et_2O. Insol. H_2O. HNO_3 ⟶ 5-nitro-6-hydroxyquinoline. $SnCl_2$ ⟶ 5-amino-6-hydroxyquinoline.

Dioxime: needles from EtOH. Decomp. at 190°.

Mathëus, *Ber.*, 1888, **21**, 1887.
v. Kostanecki, *Ber.*, 1891, **24**, 150.
v. Kostanecki, Reicher, *ibid.*, 158.

Quinoline-5 : 8-quinone



$C_9H_5O_2N$ MW, 159

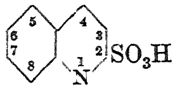
Greenish needles from EtOH. Decomp. at 110–20°. Very unstable towards alkalis. SO_2 ⟶ 5 : 8-dihydroxyquinoline.

5-*Oxime*: 5-nitroso-8-hydroxyquinoline. Needles from EtOH. Darkens at 220°. Decomp. at 245°. Prac. insol. Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O. Cold HNO_3 or $K_3Fe(CN)_6$ ⟶ 5-nitro-8-hydroxyquinoline. Hot HNO_3 ⟶ 5 : 7-dinitro-8-hydroxyquinoline. $SnCl_2$ ⟶ 5-amino-8-hydroxyquinoline.

Dioxime: cryst. from dil. EtOH. Decomp. above 200°. Sol. alkalis with yellow col.

Fischer, Renouf, *Ber.*, 1884, **17**, 1644.
v. Kostanecki, *Ber.*, 1891, **24**, 152.
v. Kostanecki, Reicher, *ibid.*, 157.

Quinoline-2-sulphonic Acid



$C_9H_7O_3NS$ MW, 209

Needles from dil. HCl. Does not melt below 270°. Spar. sol. cold H_2O. Boiling H_2O ⟶ carbostyril + SO_2. Alkali salts sol. H_2O.

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1021.

Quinoline-3-sulphonic Acid.

Needles + H_2O from H_2O. Loses H_2O at 110°. Does not melt below 270°. Mod. sol. hot H_2O.

Ba salt: needles + H_2O from H_2O. Mod. sol. H_2O.

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1032.

Quinoline-4-sulphonic Acid.

Needles from dil. HCl. Does not melt below 270°. Alkali salts sol. H_2O.

Ca salt: needles + $4H_2O$. Sol. cold H_2O.

Ag salt: needles. Sol. H_2O.

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1023.

Quinoline-5-sulphonic Acid.

Cryst. + H_2O. Sol. H_2O. Sn + HCl ⟶ 1 : 2 : 3 : 4-tetrahydroquinoline-5-sulphonic acid. Br in AcOH ⟶ 3-bromoquinoline-5-sulphonic acid. Br ⟶ 3 : 5 : 8-tribromoquinoline + 3 : 5 : 6 : 8-tetrabromoquinoline. Heat with H_2SO_4 at 250–300° ⟶ quinoline-6-sulphonic acid. KOH fusion ⟶ 5-hydroxyquinoline.

Ca salt: needles + $5H_2O$ from H_2O or dil. EtOH.

Bedall, Fischer, *Ber.*, 1882, **15**, 684.
Fischer, *Ber.*, 1882, **15**, 1979; 1887, **20**, 731.
Claus, *J. prakt. Chem.*, 1888, **37**, 258.
La Coste, Valeur, *Ber.*, 1887, **20**, 95.
Riemerschmied, *Ber.*, 1883, **16**, 721.

Quinoline-6-sulphonic Acid.

Needles + $1\frac{1}{2}H_2O$. Does not melt below 260°. Spar. sol. cold H_2O, cold EtOH. Br ⟶ 3 : 6-dibromoquinoline + 3 : 6 : 8-tribromoquinoline. KOH fusion ⟶ 6-hydroxyquinoline. KCN fusion ⟶ 6-cyanoquinoline.

Amide: m.p. 191–2°.

Happ, *Ber.*, 1884, **17**, 192.
Fischer, Willmack, *Ber.*, 1884, **17**, 440.
Knueppel, *Ber.*, 1896, **29**, 707.
v. Georgievics, *Monatsh.*, 1887, **8**, 577.

Quinoline-7-sulphonic Acid.

Needles from H_2O. Decomp. above 300°. Sol. H_2O. Spar. sol. EtOH.

Na salt: cryst. + $3H_2O$. Sol. H_2O.

K salt: cryst. + $1\frac{1}{2}$–$2H_2O$.

Ca salt: needles + $4H_2O$. Sol. H_2O.

Ba salt: cryst. + $4H_2O$.

Pb salt: cryst. powder. Sol. H_2O.

Chloride: $C_9H_6O_2NClS$. MW, 227·5. Viscous brown mass. Prac. insol. Et_2O, $CHCl_3$.

Amide: $C_9H_8O_2N_2S$. MW, 208. Cryst. M.p. 119°. Sol. EtOH, Et_2O, $CHCl_3$.

Claus, *J. prakt. Chem.*, 1888, **37**, 261.

Quinoline-8-sulphonic Acid.

Prisms. Spar. sol. H_2O. Dist. ⟶ 3 : 7′- or 4 : 7′-diquinolyl. $KMnO_4$ ⟶ quinolinic acid + 2-amino-3-sulphobenzoic acid. Sn + HCl ⟶ 1 : 2 : 3 : 4-tetrahydroquinoline-8-sulphonic acid. Br ⟶ 3 : 6 : 8-tribromoquinoline + 3 : 5 : 6 : 8-tetrabromoquinoline. KOH fusion ⟶ 8-hydroxyquinoline. H_2SO_4 at 300° ⟶ quinoline-6-sulphonic acid. Fuming HNO_3 at 160° ⟶ 8-nitroquinoline. KCN fusion ⟶ 8-cyano- + 5-cyano-quinolines.

NH_4 salt: plates + H_2O.

Na salt: needles + $5H_2O$. *Picrate*: golden leaflets from H_2O. M.p. 226–7° decomp.

K salt: prisms + $2H_2O$.

Ca salt: needles + $6H_2O$.

Ba salt: plates + $9H_2O$.

Pb salt: prisms. Spar. sol. H_2O.

Me ester: $C_{10}H_9O_3NS$. MW, 223. Prisms. M.p. 96°. Sol. EtOH, $CHCl_3$. Spar. sol. Et_2O. Prac. insol. ligroin.

Et ester: $C_{11}H_{11}O_3NS$. MW, 237. Cryst. M.p. 73°. Sol. EtOH, Et_2O, $CHCl_3$.

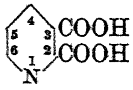
Benzyl ester: $C_{16}H_{13}O_3NS$. MW, 299. Plates or prisms from Et_2O. M.p. 84°. Sol. EtOH, $CHCl_3$. Spar. sol. ligroin.

Chloride: needles. M.p. 124° (122°). Sol. EtOH, Et_2O, $CHCl_3$.

Amide: needles from H_2O. M.p. 183–4°.

Bedall, Fischer, *Ber.*, 1882, **15**, 684.
Fischer, *Ber.*, 1882, **15**, 1979; 1887, **20**, 731.
La Coste, Valeur, *Ber.*, 1887, **20**, 95.
Claus, *J. prakt. Chem.*, 1888, **37**, 258.
v. Georgievics, *Monatsh.*, 1887, **8**, 641.
Claus, Steinitz, *Ann.*, 1894, **282**, 132.
Claus, Küttner, *Ber.*, 1886, **19**, 925.
Hoogewerff, van Dorp, *Rec. trav. chim.*, 1889, **8**, 184.
McCasland, *J. Org. Chem.*, 1946, **11**, 277.

Quinolinic Acid (*Pyridine-2 : 3-dicarboxylic acid*)



$C_7H_5O_4N$ MW, 167

Prisms from H_2O. M.p. 190–5° decomp. (rapid heat); decomp. at 110° (slow heat). Sol. 183 parts H_2O at 6·5°. Prac. insol. EtOH, Et_2O. Heat ⟶ nicotinic acid.

1-*Me ester*: $C_8H_7O_4N$. MW, 181. Prisms + H_2O from H_2O. M.p. 90°. Obtained anhyd. from AcOEt, m.p. 123°. Sol. hot H_2O, EtOH, Et_2O, $CHCl_3$, C_6H_6.

2-*Me ester*: cryst. from C_6H_6. M.p. 106°. Sol. H_2O and most org. solvents.

1-*Me ester* 2-*chloride*: $C_8H_6O_3NCl$. MW, 199·5. Needles. M.p. 126°.

Di-Me ester: $C_9H_9O_4N$. MW, 195. Plates from CS_2–ligroin. M.p. 54–5°.

1-*Et ester*: $C_9H_9O_4N$. MW, 195. Plates from C_6H_6. M.p. 132°. Also prisms + H_2O from H_2O.

1-*Et ester* 2-*chloride*: $C_9H_8O_3NCl$. MW, 213·5. M.p. 163°.

1-*Me* 2-*Et ester*: $C_{10}H_{11}O_4N$. MW, 209. B.p. 250–5° decomp. B_2,H_2PtCl_6: orange-red cryst. from alc. HCl. M.p. 165° decomp.

2-*Me* 1-*Et ester*: b.p. 254–8° decomp. B_2,H_2PtCl_6: yellow needles from alc. HCl. M.p. 174° decomp.

Di-Et ester: $C_{11}H_{13}O_4N$. MW, 223. B.p. 280–5° part decomp., 170°/15 mm.

Dichloride: $C_7H_3O_2NCl_2$. MW, 204. B.p. 159°/19 mm.

1-*Amide*: $C_7H_6O_3N_2$. MW, 166. Prisms. M.p. 168·5° decomp. Spar. sol. cold H_2O.

2-*Amide*: prisms. M.p. 160° decomp.

Diamide: $C_7H_7O_2N_3$. MW, 165. Needles from EtOH. M.p. 209° decomp. (190° decomp.). Sol. H_2O, EtOH.

Imide: $C_7H_4O_2N_2$. MW, 148. Needles from EtOH. M.p. 233° (230°). Spar. sol. H_2O, EtOH, AcOH. Insol. C_6H_6, ligroin.

Anhydride: $C_7H_3O_3N$. MW, 149. M.p. 161–2°.

3-*Nitrile*: 3-cyanopicolinic acid. $C_7H_4O_2N_2$. MW, 148. Needles from H_2O. M.p. 175–6°. Sol. EtOH. Spar. sol. Et_2O, $CHCl_3$, pet. ether.

Dihydrazide: needles from EtOH. M.p. 224°. Sol. H_2O. Spar. sol. EtOH.

Me-betaine: plates + H_2O. M.p. 151° decomp. Sol. warm H_2O.

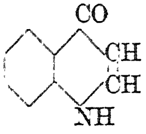
Hoogewerff, van Dorp, *Ber.*, 1879, **12**, 747; *Ann.*, 1880, **204**, 87, 116.
Phillips, *Ann.*, 1895, **288**, 255.
Scheiber, Knothe, *Ber.*, 1912, **45**, 2256.
Kirpal, *Monatsh.*, 1900, **21**, 959; 1901, **22**, 361.
Meyer, *Monatsh.*, 1901, **22**, 580.
Engler, *Ber.*, 1894, **27**, 1787.
Stix, Bulgatsch, *Ber.*, 1932, **65**, 11.
Sucharda, *Ber.*, 1925, **58**, 1727.
Kulka, *J. Am. Chem. Soc.*, 1946, **68**, 2472.
Kamp, Sletzinger, B.P. 596,230, (*Chem. Abstracts*, 1948, **42**, 3783).

9*H*-Quinolizine.

See Pyridocoline.

α-Quinolone.
See Carbostyril.

γ-Quinolone (*Keto form of 4-hydroxyquinoline*)



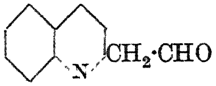
C_9H_7ON MW, 145

Cryst. from EtOH. M.p. 235°.
Acetyl: m.p. 228°.
Phenylhydrazone: needles from dil. EtOH. M.p. 168°.
N-*Me*: see Echinopsine.
N-*Et*: dark resin. Sol. H_2O. Prac. insol. Et_2O. *Chloroaurate*: m.p. 155° decomp.

Reissert, *Ber.*, 1887, **20**, 3109; 1888, **21**, 1376.
Meyer, *Monatsh.*, 1906, **27**, 265.

γ-Quinolone-2-carboxylic Acid (*Keto form of kynurenic acid*).
The compound (m.p. 167°) regarded as γ-quinolone-2-carboxylic acid by Heller and Sourlis (*Ber.*, 1908, **41**, 2699) has been shown by Meisenheimer and Stotz (*Ber.*, 1925, **58**, 2334) to be quinaldinic acid *N*-oxide.

2-Quinolylacetaldehyde

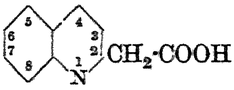


$C_{11}H_9ON$ MW, 171

Cryst. from H_2O or EtOH. M.p. 184° (103–4°). Sol. acids. Reduces $NH_3.AgNO_3$.
Oxime: m.p. 205°.
Phenylhydrazone: yellow needles from EtOH. M.p. 198–9°.
B_2,H_2PtCl_6: yellow cryst. + $2H_2O$ from dil. HCl.
Semicarbazone: needles from EtOH. M.p. 244° decomp.
Picrate: yellow cryst. from dil. EtOH. M.p. 212°.

Einhorn, *Ber.*, 1886, **19**, 908.
Einhorn, Sherman, *Ann.*, 1895, **287**, 38.
Hupe, Schramme, *Z. physiol. Chem.*, 1928, **177**, 315.
Kenner, Nandi, *Ber.*, 1936, **69**, 639.
Borsche, Manteuffel, *Ann.*, 1936, **526**, 45.

2-Quinolylacetic Acid



$C_{11}H_9O_2N$ MW, 187

Prisms from MeOH. M.p. 274–5° (271–2°). Sublimes. Ca salt insol. H_2O, EtOH.
Me ester: $C_{12}H_{11}O_2N$. MW, 201. Flakes from ligroin. M.p. 72°.
Et ester: $C_{13}H_{13}O_2N$. MW, 215. Plates from ligroin. M.p. 68–9° (67°). B.p. 240°/16 mm. *Picrate*: m.p. 152°.
Nitrile: $C_{11}H_{10}ON_2$. MW, 186. M.p. 53–4°. B.p. 140°/1 mm. *Picrate*: m.p. 176–7° decomp.
B_2,H_2PtCl_6: dimorphous. Needles and brownish-red plates. Sol. H_2O.
Picrate: yellow needles from MeOH. M.p. 236–7°.

Einhorn, Sherman, *Ann.*, 1895, **287**, 40.
Kenner, Nandi, *Ber.*, 1936, **69**, 639.
Borsche, Manteuffel, *Ann.*, 1936, **526**, 22.

3-Quinolylacetic Acid.
Free acid not described.
Et ester: $C_{13}H_{13}O_2N$. Cryst. from pet. ether. M.p. 84°. B.p. 140–2°/2·5 mm.

Jones, Soper, Behrens, Corse, *J. Am. Chem. Soc.*, 1948, **70**, 2843.

4-Quinolylacetic Acid.
Decomp. at 90° to lepidine + CO_2.
Et ester: m.p. 64°.
Nitrile: m.p. 144–5°.

Borsche, Bütschli, *Ann.*, 1937, **529**, 266.

6-Quinolylacetic Acid.
M.p. 218–20° decomp.
Et ester: b.p. 160°/3 mm.

Jones, Soper, Behrens, Corse, *J. Am. Chem. Soc.*, 1948, **70**, 2843.

8-Quinolylacetic Acid.
Free acid not described.
Et ester: b.p. 158–60°/3 mm.
Nitrile: $C_{11}H_8N_2$. MW, 168. Cryst. M.p. 88°.

Claus, D.R.P., 98,272, (*Chem. Zentr.*, 1898, II, 744).
Howitz, Nöther, *Ber.*, 1906, **39**, 2706.
Jones, Soper, Behrens, Corse, *J. Am. Chem. Soc.*, 1948, **70**, 2843.

2-[2-Quinolyl]-acrylic Acid

$C_{12}H_9O_2N$ MW, 199

Plates from MeOH. Decomp. at 194–6° (190–5°). $KMnO_4$ (according to conditions) ⟶ either quinoline-2-aldehyde or 1 : 2-dihydroxy-2-[2-quinolyl]-propionic acid. $SnCl_2$ or NaHg ⟶ 2-[2-quinolyl]-propionic acid. Na + EtOH or Sn + HCl ⟶ 2-[2-(1 : 2 : 3 : 4-tetrahydroquinolyl)]-propionic acid.
Et ester: $C_{14}H_{13}O_2N$. MW, 227. Needles from ligroin. M.p. 73°.
Amide: $C_{12}H_{10}ON_2$. MW, 198. Needles from H_2O. M.p. 175–6°. Sol. warm. EtOH, C_6H_6.
B,HCl: m.p. 216–18°.
B,HBr: m.p. 218–20°.

Acetate: m.p. 203°.

Einhorn, *Ber.*, 1886, **19**, 908.
Einhorn, Sherman, *Ann.*, 1895, **287**, 27.
v. Miller, Spady, *Ber.*, 1885, **18**, 3403; 1886, **19**, 130.
Alberts, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1285.

2-[4-Quinolyl]-acrylic Acid.

Needles from EtOH–AcOH. M.p. 272–3° decomp. Spar. sol. EtOH. Prac. insol. H_2O. HI(+ P) in AcOH ⟶ 2-[4-quinolyl]-propionic acid.

Et ester, HCl: m.p. 194–5°.

B_2,H_2PtCl_6: yellow needles + $1\frac{1}{2}H_2O$ from H_2O.

Koenigs, Müller, *Ber.*, 1904, **37**, 1338.
Clemo, Hoggarth, *J. Chem. Soc.*, 1939, 1241.
Phillips, *J. Am. Chem. Soc.*, 1948, **70**, 452.

Quinolylamine.

See Aminoquinoline.

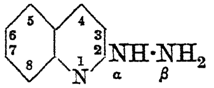
α-Quinolylcarbinol.

See 2-Hydroxymethyl-quinoline.

Quinolylethylene.

See Vinylquinoline.

2-Quinolylhydrazine (2-*Hydrazinoquinoline*)



$C_9H_9N_3$ MW, 159

Cryst. from C_6H_6. M.p. 142–3° (134–5°). Sol. EtOH. Spar. sol. Et_2O, ligroin.

B_2,H_2PtCl_6: cryst. ppt. M.p. 170° decomp.

Picrate: m.p. 187° decomp.

β-N-*Phenyl*: *sym.*-phenylquinolylhydrazine. Needles from EtOH. M.p. 191°. Unstable. Sol. $CHCl_3$, AcOH. Spar. sol. EtOH. Prac. insol. Et_2O.

β-N-*Acetyl*: prisms from EtOH. M.p. 195°.

β-N-*Benzoyl*: needles from Me_2CO. M.p. 204°.

Perkin, Robinson, *J. Chem. Soc.*, 1913, **103**, 1978.
Marckwald, Meyer, *Ber.*, 1900, **33**, 1885.
Ephraim, *Ber.*, 1891, **24**, 2818.
Fargher, Furness, *J. Chem. Soc.*, 1915, **107**, 697.

5-Quinolylhydrazine.

Yellow needles from H_2O. M.p. 150–1°. Sol. EtOH. Spar. sol. C_6H_6. Insol. pet. ether.

B,2HCl: yellow needles. M.p. 248°.

Dufton, *J. Chem. Soc.*, 1892, **61**, 785.

6-Quinolylhydrazine.

Non-crystalline. Readily resinifies.

B,HCl: cryst. from dil. EtOH.

Benzylidene deriv.: yellowish-red needles from H_2O or red cryst. from EtOH. M.p. 203°.

Knueppel, *Ann.*, 1900, **310**, 82.

8-Quinolylhydrazine.

Needles. M.p. 64°.

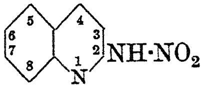
B,2HCl: yellow prisms.

Dufton, *J. Chem. Soc.*, 1891, **59**, 757.

2-Quinolyl Mercaptan.

See Thiocarbostyril.

2-Quinolylnitramine (2-*Nitraminoquinoline*, N-*nitro*-2-*aminoquinoline*)



$C_9H_7O_2N_3$ MW, 189

Yellowish needles from AcOH. M.p. 223–5° decomp. Turns red in air.

Tschitschibabin, Witkovsky, Lapschin, *Ber.*, 1925, **58**, 806.

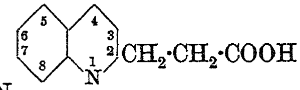
4-Quinolylnitramine (4-*Nitraminoquinoline*, N-*nitro*-4-*aminoquinoline*).

Yellow needles + H_2O from H_2O. M.p. 207° decomp. Sol. EtOH, hot H_2O.

B_2,H_2PtCl_6: orange-red cryst. Decomp. at 210°. Hyd. by H_2O.

Tschitschibabin, Witkovsky, Lapachin, *Ber.*, 1925, **58**, 806.
Claus, Frobenius, *J. prakt. Chem.*, 1897, **56**, 202.

2-[2-Quinolyl]-propionic Acid



$C_{12}H_{11}O_2N$ MW, 201

Plates from C_6H_6. M.p. 122–3°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, Me_2CO. Insol. H_2O, ligroin.

Amide: $C_{12}H_{12}ON_2$. MW, 200. Needles from C_6H_6. M.p. 149–50°. Sol. hot H_2O, EtOH, C_6H_6. Insol. pet. ether.

Hydrazide: m.p. 165°. Reduces Fehling's and $NH_3.AgNO_3$.

B_2,H_2PtCl_6: brownish-red plates from dil. HCl. M.p. 197° decomp.

Einhorn, Sherman, *Ann.*, 1895, **287**, 29.
Koenigs, *Ber.*, 1900, **33**, 220.
Kermack, Muir, *J. Chem. Soc.*, 1931, 3092.

2-[4-Quinolyl]-propionic Acid.

Needles from H_2O. M.p. 202–3°. Sol. EtOH, hot H_2O, boiling Me_2CO. Na + EtOH ⟶ 2-[4-(1 : 2 : 3 : 4-tetrahydroquinolyl)]-propionic acid.

Koenigs, Müller, *Ber.*, 1904, **37**, 1339.

Quinone.

See *p*-Benzoquinone.

Quinone-anilide.

See 2 : 5-Dianilino-*p*-benzoquinone.

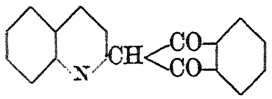
Quinophan.

See 2-Phenylquinoline-4-carboxylic Acid.

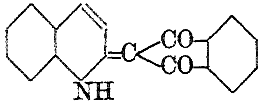
Quinophenol.

See 8-Hydroxyquinoline.

Quinophthalone



or



$C_{18}H_{11}O_2N$ MW, 273

Golden-yellow needles from EtOH. M.p. 241° (234°). Sol. hot AcOH, hot Me_2CO, $CHCl_3$, warm C_6H_6, toluene. Prac. insol. EtOH, Et_2O. Sol. conc. HCl and conc. H_2SO_4 with red col. Sublimes. Warm HNO_3 (D 1·2) ⟶ quinaldinic + phthalic acids.

Phenylhydrazone: red needles from $CHCl_3$. M.p. 206°.

Anil: red needles from EtOH. M.p. 232°. Sol. $CHCl_3$. Spar sol. EtOH.

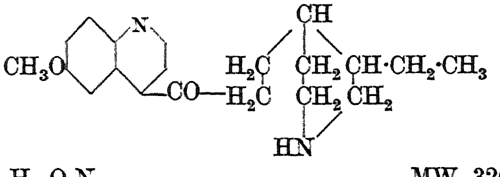
N-Me: orange-yellow needles or brownish-red prisms. M.p. 249·5°.

Jacobsen, Reimer, *Ber.*, 1883, **16**, 1082.
Eibner, *Ber.*, 1904, **37**, 3606.
Eibner, Lange, *Ann.*, 1901, **315**, 336.
Eibner, Hofmann, *Ber.*, 1904, **37**, 3015, 3018.
Kuhri, Bär, *Ann.*, 1935, **516**, 155.
Weno, Suzuki, *J. Soc. Chem. Ind. Japan*, 1933, **36**, Supplementary binding, 195.

Quinosol.

See under 8-Hydroxyquinoline.

Quinoticine (*Quinotoxine dihydride, quinicine dihydride, hydroquinotoxine, hydroquinicine*)



$C_{20}H_{26}O_2N_2$ MW, 326

Yellow cryst. Sol. EtOH, Et_2O, $CHCl_3$, dil. acids. $[\alpha]_D^{15}$ − 17° in HCl. Gives yellow col. in H_2SO_4 but no fluor.

B_2,H_2PtCl_6: orange cryst. + H_2O. Insol. H_2O.

Hesse, *Ann.*, 1887, **241**, 273.
Rabe, Kindler, *Ber.*, 1919, **52**, 1844.
See also Kaufmann, Rothlin, Brunnschweiler, *Ber.*, 1916, **49**, 2303.

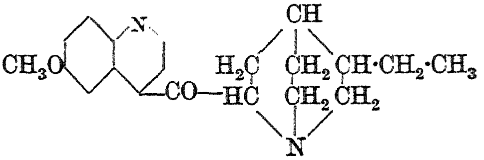
Quinotidine.

See Hydroquinidine.

Quinotine.

See Hydroquinine.

Quinotinone (*Quininone dihydride, hydroquininone*)



$C_{20}H_{24}O_2N_2$ MW, 324

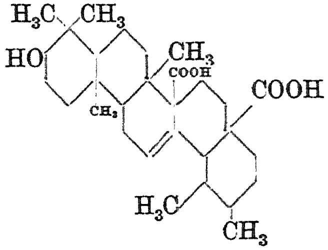
M.p. 100°. $[\alpha]_D^{19}$ + 73·15° in EtOH, $[\alpha]_D^{20}$ + 65·0° in 96% EtOH.

Rabe, Kindler, *Ber.*, 1919, **52**, 1845.
See also Kaufmann, Rothlin, Brunnschweiler, *Ber.*, 1916, **49**, 2303.

Quinotoxine.

See Quinicine.

Quinovaic Acid



Suggested structure

$C_{30}H_{46}O_5$ MW, 486

Needles. M.p. 298°. Spar. sol. hot EtOH, AcOH. Insol. H_2O. Sol. NH_3 and alkalis. $[\alpha]_D$ + 87–8° in KOH. Decomp. at 300° ⟶ pyroquinovaic acid. Sol. H_2SO_4 with evolution of CO.

Di-Me ester: $C_{32}H_{50}O_5$. MW, 514. M.p. 173–4°. *Acetyl*: m.p. 208–9°. *Benzoyl*: m.p. 235–6°.

Di-Et ester: $C_{34}H_{54}O_5$. MW, 542. Cryst. M.p. 127–30°. Sol. EtOH, Et_2O.

Acetyl: m.p. 284° decomp.

Benzoyl: m.p. 284° decomp.

Jeger, *Über die Konstitution der Triterpene, Fortschritte der Chemie organischer Naturstoffe*, Vol. VII, 1950. (Springer-Verlag, Vienna).
Brossi, Bischof, Jeger, Ruzicka, *Helv. Chim. Acta*, 1951, **34**, 244.

Quinovin

$C_{36}H_{56}O_9$ MW, 632

α-.

Occurs with cinchona alkaloids as glycoside. Cryst. powder or needles from EtOH. Sol. EtOH. Spar. sol. Et_2O, $CHCl_3$, C_6H_6. Prac. insol. H_2O. $[\alpha]_D$ + 59·1° (+ 56·6°) in EtOH. Bitter taste. Sol. NH_3 and alkalis. Sol. conc. H_2SO_4 with orange-yellow col. and evolution of CO. Reduces Fehling's. Hyd. ⟶ quinovaic acid + quinovose (isorhodeose).

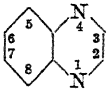
β-.
Occurs with alkaloids from the *Remijia* species. Plates from dil. EtOH. M.p. 235° decomp. Sol. EtOH. Insol. Et_2O, AcOEt. $[\alpha]_D + 27{\cdot}9°$ in EtOH.

Hlasiwetz, *Ann.*, 1859, **111**, 182.
Rochleder, *J. prakt. Chem.*, 1867, **102**, 16.
Liebermann, Giesel, *Ber.*, 1883, **16**, 928.

Quinovose (*Chinovose*).
This sugar has been shown to be identical with isorhodeose, (*q.v.*).

See Votoček, Rác, *Chem. Abstracts*, 1929, **23**, 4449; 1932, **26**, 4307.

Quinoxaline (*Benzpyrazine, phenpiazine*)



$C_8H_6N_2$ MW, 130

Cryst. (anhyd.) from pet. ether. M.p. 28°. Cryst. ($+ H_2O$) by pptn. from pet. ether by addn. of H_2O, m.p. 37°. On standing in air anhyd. cryst. ⟶ monohydrate. B.p. 225–6° (220–3°, 229·5°/760·3 mm.), 140°/40 mm. D_4^{48} 1·1334. n_D^{48} 1·6231. Sol. H_2O, EtOH, Et_2O, C_6H_6. $KMnO_4$ ⟶ pyrazine-2 : 3-dicarboxylic acid. Na + EtOH ⟶ 1 : 2 : 3 : 4-tetrahydroquinoxaline.
B,HCl: needles. Sinters at 170°. Decomp. at 184°. Sol. H_2O, EtOH.
B,H_2SO_4: plates. M.p. 186–7°. Sol. H_2O. Mod. sol. EtOH.
B_2,H_2PtCl_6: yellow needles.
Oxalate: needles. M.p. 169°. Spar. sol. H_2O.
Methiodide: reddish-yellow plates from EtOH. M.p. 176° decomp. Sol. H_2O. Mod. sol. EtOH.
Ethiodide: red needles from EtOH. M.p. 146° decomp. Sol. H_2O.

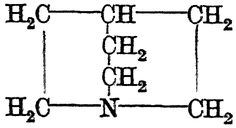
Hinsberg, *Ber.*, 1884, **17**, 320; *Ann.*, 1887, **237**, 334; 1896, **292**, 245.
Merz, Ris, *Ber.*, 1887, **20**, 1194.

Quinoxyl.
See Yatren.

Quinpropyline.
See under Cupreine.

***p*-Quinquiphenyl.**
See p-Pentaphenyl.

Quinuclidine (*Nuclidine, 1 : 4-ethylenepiperidine*)



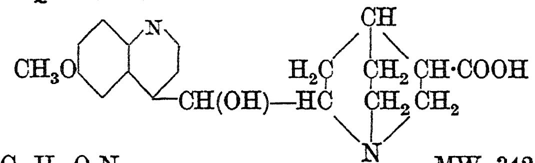
$C_7H_{13}N$ MW, 111

Cryst. M.p. 158° (sealed tube). Very sol. H_2O, org. solvents. Very volatile. Ammoniacal odour. Does not decolourise $KMnO_4$.

$B,HAuCl_4$: leaflets from H_2O. M.p. 271–3° decomp.
B_2,H_2PtCl_6: leaflets from H_2O. M.p. 238–40° decomp.
Ethiodide: plates from EtOH. M.p. 270–1°. Slightly hygroscopic.
Ethochloroplatinate: leaflets from hot H_2O. M.p. 271–2° (212°) decomp.
Picrate: pale yellow needles from EtOH. M.p. 275–6°. Sol. 35–40 parts hot EtOH.

Meisenheimer, Neresheimer, Schneider, *Ann.*, 1920, **420**, 213.
Prelog *et al.*, *Ann.*, 1937, **532**, 69.
Prelog, Cerkovnikov, Ustricev, *Ann.*, 1938, **535**, 37.
Clemo, Metcalfe, *J. Chem. Soc.*, 1937, 1989.

Quitenine

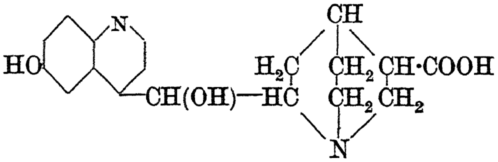


$C_{19}H_{22}O_4N_2$ MW, 342

Prisms $+ 4H_2O$ from dil. EtOH. M.p. 286° decomp. (228° anhyd.). Loses H_2O at 110°. Sol. acids and alkalis. Spar. sol. H_2O. Insol. Et_2O. Sol. in dil. H_2SO_4 shows blue fluor. $[\alpha]_D - 143°$ in EtOH. Heat with H_2SO_4 ⟶ quitenicine. HI ⟶ quitenol + CH_3I. Gives thalleioquin reaction.
$B_3,2H_2SO_4$: prisms $+ 15H_2O$. Sol. H_2O. Spar. sol. EtOH.
B,H_2PtCl_6: yellow leaflets $+ 3H_2O$. Spar. sol. H_2O.
Et ester: needles. M.p. 198°. Insol. H_2O.
Ethiodide: prisms. M.p. 210°.

Skraup, *Ann.*, 1879, **199**, 348.
Bucher, *Monatsh.*, 1893, **14**, 598.
Giemsa, Oesterlin, *Ber.*, 1931, **64**, 60.

Quitenol



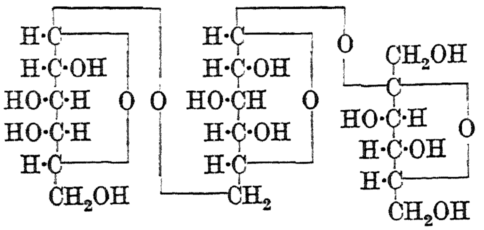
$C_{18}H_{20}O_4N_2$ MW, 328

Needles $+ H_2O$. Decomp. above 270° without melting. Sol. acids, alkalis. Pptd. from alk. sol. by CO_2. Prac. insol. H_2O, EtOH, Et_2O. $FeCl_3$ in dil. HCl gives red col. Gives thalleioquin reaction.
B,2HCl: plates $+ H_2O$.
B_2,H_2PtCl_6: yellow prisms.
B,H_2SO_4: yellow plates $+ H_2O$.
Me ether: *see* Quitenine.

Bucher, *Monatsh.*, 1893, **14**, 604.

R

Racemic Acid.
See Tartaric Acid.
R-Acid.
See 2-Naphthol-3 : 6-disulphonic Acid.
2-R Acid.
See 2-Amino-8-naphthol-3 : 6-disulphonic Acid.
Raffinose (*Melitriose, gossypose*)



$C_{18}H_{32}O_{16}$ MW, 504

Occurs in sugar beet, cotton seed, manna, etc. White needles or prisms + $5H_2O$. M.p. 80°, anhyd. 118–19°. Slight sweet taste. $[\alpha]_D$ + 104° in H_2O (hydrate), + 123° in H_2O (anhyd.). D^0 1·465. Sol. H_2O. Mod. sol. MeOH. Insol. EtOH, Et_2O. Does not reduce Fehling's. Does not form an osazone. Hyd. by dil. acids ⟶ galactose + glucose + fructose. Emulsin ⟶ sucrose + galactose. Invertase or raffinase ⟶ melibiose + fructose. HNO_3 ⟶ saccharic and mucic acids.

Undeca-acetyl: cryst. from EtOH. M.p. 99–101°. $[\alpha]_D$ + 92·2°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. CS_2, ligroin.

Octabenzoyl: cryst. from AcOH. M.p. 98°. $[\alpha]_D^{18\cdot5}$ + 155·3° in AcOH.

Undecabenzoyl: m.p. 113°. $[\alpha]_D^{20\cdot5}$ + 106·8°. Sol. $CHCl_3$, AcOEt. Mod. sol. Et_2O, Me_2CO, C_6H_6, hot MeOH, hot EtOH.

Undeca-p-*chlorobenzoyl*: m.p. 130–2°. $[\alpha]_D^{18}$ + 96·8°.

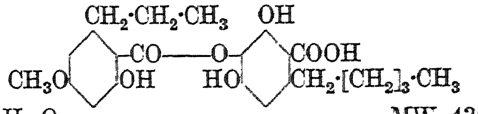
Undeca-p-*bromobenzoyl*: m.p. 138°. $[\alpha]_D^{20\cdot5}$ + 85·2°.

Undeca-Me ether: b.p. 238–40°/0·02 mm.

Harding, *Sugar*, 1923, **25**, 308.
Englis, Decker, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2724.
Hudson, Harding, *J. Am. Chem. Soc.*, 1914, **36**, 2110.
Vogel, Pictet, *Helv. Chim. Acta*, 1928, **11**, 898.
Haworth, Charlton, Hickinbottom, *J. Chem. Soc.*, 1927, 1527.
Hungerford, Nees, *Ind. Eng. Chem.*, 1934, **26**, 462.

Ramalic Acid.
See Obtusatic Acid.

Ramalinolic Acid



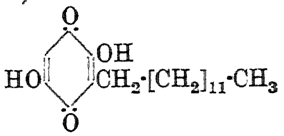
$C_{23}H_{28}O_8$ MW, 432

Prisms from C_6H_6. M.p. 164°. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. C_6H_6, $CHCl_3$. $FeCl_3$ ⟶ violet col. in EtOH.

Tri-Me ether Me ester: $C_{27}H_{36}O_8$. MW, 488. M.p. 75°.

Asahina, Kusaka, *Ber.*, 1936, **69**, 1896.

Rapanone (2 : 5-*Dihydroxy-3-tridecyl*-1 : 4-*benzoquinone*)



$C_{19}H_{30}O_4$ MW, 322

Anthelmintic agent from *Embelia ribes*. Glistening golden orange plates from EtOH. M.p. 141–2°.

Dibenzoyl: light yellow needles. M.p. 88–90°.

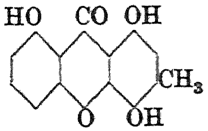
Mono-Me ether: m.p. 95°.

Tetra-acetyl deriv.: small colourless needles. M.p. 116–17°.

Kuroda, *Chem. Abstracts*, 1939, **33**, 8600.
Asano, Yamaguti, *Chem. Abstracts*, 1942, **36**, 81.
Fieser, Chamberlain, *J. Am. Chem. Soc.*, 1948, **70**, 71.

Ratanhin.
See Surinamine.

Ravenelin (1 : 4 : 5-*Trihydroxy-2-methylxanthone*)



$C_{14}H_{10}O_5$ MW, 258

Found in *Helminthosporium Ravenlii*, C., and *H. Turcicum*, P. Yellow prismatic needles from Me_2CO–$CHCl_3$. M.p. 267–8°. Sol. Py, hot AcOH. Mod. sol. hot EtOH, Me_2CO. Spar. sol. C_6H_6, pet. ether. Hot Na_2CO_3.Aq. ⟶ brownish-yellow sol. NaOH ⟶ yellow sol. ⟶ deep brown on shaking in air. Alc. $FeCl_3$ ⟶ intense greenish-brown col. Conc. H_2SO_4 ⟶ orange-yellow sol. ⟶ orange-brown on heating.

Di-Me ether: $C_{16}H_{14}O_5$. MW, 286. Yellow needles from EtOH. M.p. 285–7°.

Tri-Me ether: $C_{17}H_{16}O_5$. MW, 300. Prisms from EtOH. M.p. 178–9°. Conc. HCl ⟶ intense orange sol. *B,HCl,FeCl₃*: red prisms from AcOH. M.p. 174–5°.

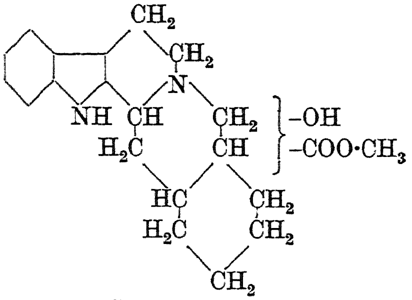
Triacetyl: needles from AcOH. M.p. 204–5°. Sol. hot EtOH, C_6H_6.

Tribenzoyl: prismatic needles from Py. M.p. 255°. Sol. warm Py. Mod. sol. hot AcOH. Spar. sol. boiling EtOH.

Trianisoyl: needles from AcOH. M.p. 216–18°.

Raistrick, Robinson, White, *Biochem. J.*, 1936, **30**, 1303.

Rauwolscine



Suggested structure

$C_{21}H_{26}O_3N_2$ MW, 354

Occurs in *R. Canescens*. Thick pale yellow plates from C_6H_6. M.p. 231–2° decomp. $[\alpha]_D^{30}$ $-40°$ in EtOH. Sol. Et_2O, AcOEt, $CHCl_3$. Spar. sol. H_2O, pet. ether.

B,HCl: colourless prismatic needles from hot H_2O. M.p. 278–80° decomp. $[\alpha]_D^{30}$ $+74°$ in H_2O.

B,HNO₃: prismatic needles from hot H_2O. M.p. 257–8° decomp.

B,H₂SO₄: colourless needles from EtOH. M.p. 256–7° decomp.

B,H₂PtCl₆: amorph. orange needles from H_2O. M.p. 255–7° decomp.

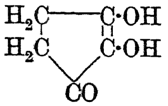
Picrate: shining orange plates from EtOH. M.p. 208° decomp.

Mookerjee, *J. Indian Chem. Soc.*, 1946, **23**, 6, (*Chem. Abstracts*, 1946, **40**, 5750).

R.D.X.

See Cyclonite.

Reductic Acid (1 : 2-*Dihydroxycyclopentenone*-3)



$C_5H_6O_3$ MW, 114

Yellow cryst. from AcOEt. M.p. 213–213·5° decomp. (207·5°). Sol. H_2O, EtOH, MeOH. Spar. sol. Et_2O, AcOEt, Me_2CO. Insol. C_6H_6.

Acetyl deriv.: m.p. 195°.

Diacetyl deriv.: oil. B.p. 112°/0·2 mm.

Anilide: yellow needles. M.p. 197°.

Osazone: reddish-brown needles. M.p. 246–7°.

Me ether: $C_6H_8O_3$. MW, 128. Needles. M.p. 138°.

Di-Me ether: $C_7H_{10}O_3$. MW, 142. B.p. 120°/12 mm.

Llgami, *Chem. Abstracts*, 1934, **28**, 7259.
Reichstein, Oppenauer, *Helv. Chim. Acta*, 1934, **17**, 390.
Aso, *Chem. Abstracts*, 1940, **34**, 379.
Hesse, Bücking, *Ann.*, 1949, **563**, 31.

Reductone (*Dihydroxyacrolein*)

HO·CH:C(OH)·CHO

$C_3H_4O_3$ MW, 88

Needles from H_2O. Darkens at 150–70°. Decomp. at 200–20°. MeOH–$Ba(OH)_2$ ⟶ Ba salt. Sodium methoxide ⟶ Na salt. Ox. with I to a tricarbonyl comp.

Na salt: fine cryst. ppt. Insol. EtOH.

Ba salt: fine cryst. ppt. Insol. EtOH.

Mono-Me ether: colourless leaflets from Et_2O at $-80°$. M.p. 67°. Alc. $FeCl_3$ ⟶ dark blue col.

Mono-phenylhydrazone of ox. product: red cryst. from EtOH. M.p. 176°. Conc. H_2SO_4 ⟶ blue-violet col.

Di-phenylhydrazone of ox. product: yellow needles from EtOH. M.p. 166°. Conc. H_2SO_4 ⟶ green col.

Euler, Martius, *Ann.*, 1933, **505**, 73.

Regularobufagin

$C_{25}H_{34}O_6$ MW, 430

Prisms from EtOH.Aq. M.p. 235–6°.

Diacetyl deriv.: m.p. 224–5° (196–7°).

Kotake, *J. Chem. Soc. Japan*, 1934, **55**, 179.
Jensen, *J. Am. Chem. Soc.*, 1935, **57**, 1765.

Reichstein's Substance Fa.

See Cortisone.

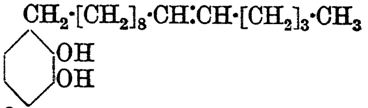
Reichstein's Substance H.

See Corticosterone.

Remerine.

See Roemerine.

Renghol (*Dihydrourushiol*, 15-[2:3-*dihydroxyphenyl*]-5-*pentadecylene*)



$C_{21}H_{34}O_2$ MW, 318

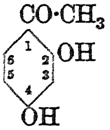
Toxic constituent of renghas fruit (*Semecarpus heterophylla*, Bl.). M.p. 14–15°. B.p. 170–2°/0·0001 mm. Ozonolysis ⟶ valeraldehyde. Vesicant.

Me ether: $C_{22}H_{36}O_2$. MW, 332. B.p. 160°/0·0001 mm.

Di-Me ether: $C_{23}H_{38}O_2$. MW, 346. B.p. 226°/5 mm., 155–7°/0·0001 mm.

Backer, Haack, *Rec. trav. chim.*, 1938, **57**, 225.

Resacetophenone (*4-Acetoresorcinol, 2 : 4-dihydroxyacetophenone*)



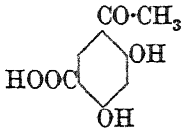
$C_8H_8O_3$ MW, 152

Leaflets or needles. M.p. 147° (145–6°). $FeCl_3 \longrightarrow$ red col.

2-Acetyl: m.p. 119–20°.
4-Acetyl: m.p. 74°. *2-Benzoyl*: m.p. 67°.
Diacetyl: m.p. 38°.
4-Benzoyl: m.p. 106–7°. *2-Anisoyl*: m.p. 109–10°.
Dibenzoyl: m.p. 80–1°.
Dianisoyl: m.p. 118°.
Diveratroyl: m.p. 151–2°.
Dicinnamoyl: m.p. 131°.
Oxime: m.p. 198–200° decomp. *Acetyl*: m.p 174–5° decomp.
Semicarbazone: m.p. 214–20° decomp.
Phenylhydrazone: m.p. 159°.
Dinitrophenylhydrazone: maroon cryst. M.p. 242–5°.
2-Me ether: *see* Isopæonol (Isopeonol).
4-Me ether: *see* Peonol.
Di-Me ether: 2 : 4-dimethoxyacetophenone, peonol methyl ether, isopeonol methyl ether. $C_{10}H_{12}O_3$. MW, 180. M.p. 44° (40°). *Oxime*: m.p. 125°.
2-Me: *4-Et ether*: isopeonol ethyl ether. $C_{11}H_{14}O_3$. MW, 194. Needles from EtOH. M.p. 49°.
4-Me: *2-Et ether*: peonol ethyl ether. Needles from EtOH. M.p. 70°. $D^{80.2}$ 1·0571. $n_{\alpha}^{80.2}$ 1·51434.
4-Et ether: $C_{10}H_{12}O_3$. MW, 180. Needles from EtOH. M.p. 49°.
Di-Et ether: 2 : 4-diethoxyacetophenone. $C_{12}H_{16}O_3$. MW, 208. M.p. 74°. *Oxime*: m.p. 122°.
4-Propyl ether: $C_{11}H_{14}O_3$. MW, 194. M.p. 25°.
Dipropyl ether: $C_{14}H_{20}O_3$. MW, 236. M.p. 26°.
4-Butyl ether: $C_{12}H_{16}O_3$. MW, 208. M.p. 43°.
Dibutyl ether: $C_{16}H_{24}O_3$. MW, 264. M.p. 32°.
4-α-Glycerol ether: $C_{11}H_{14}O_5$. MW, 226. M.p. 88°. *Phenylhydrazone*: m.p. 119–20°.
4-Glucoside: cryst. M.p. 201–2°. *Tetra-acetyl*: m.p. 131–2°.

Robinson, Shah, *J. Chem. Soc.*, 1934, 1494.
Gulati, Venkataraman, *J. Chem. Soc.*, 1931, 2376.
Kostanecki, Tambor, *Ber.*, 1895, **28**, 2306.
Schaffer, U.S.P., 1,745,507, (*Chem. Abstracts*, 1930, **24**, 1707).
Kondo, Nakagawa, *Chem. Abstracts*, 1931, **25**, 515.
Cox, *Rec. trav. chim.*, 1931, **50**, 850.
Baker, *J. Chem. Soc.*, 1934, 1691.
Nadkarni, Wheeler, *J. Chem. Soc.*, 1936, 589.
Mauthner, *J. prakt. Chem.*, 1942, **160**, 33; 1943, **161**, 284.
Cooper, *Organic Syntheses*, 1941, XXI, 103.

Resacetophenone-5-carboxylic Acid (*2 : 4-Dihydroxyacetophenone-5-carboxylic acid, 2 : 4-dihydroxy-5-acetobenzoic acid*)

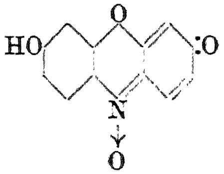


$C_9H_8O_5$ MW, 196

Needles from Me_2CO. M.p. 256° decomp.
Me ester: $C_{10}H_{10}O_5$. MW, 210. Needles from C_6H_6. M.p. 124–5°. *Hydrazone*: (i) m.p. 138°. (ii) M.p. 170°.
Di-Me ether: $C_{11}H_{12}O_5$. MW, 224. Prisms from MeOH. M.p. 231–3°.
Et ester: $C_{11}H_{12}O_5$. MW, 224. M.p. 94°.
Di-Et ether: $C_{15}H_{20}O_5$. MW, 280. Needles from ligroin. M.p. 96–7°.
p-*Bromophenylhydrazone*: m.p. 243°.

Liebermann, Lindenbaum, *Ber.*, 1908, **41**, 1610.

Resazurin



$C_{12}H_7O_4N$ MW, 229

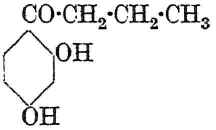
Small prisms from AcOH. Blue col. Red. by bacteria in milk $\longrightarrow$ resorufin (pink col.). Used for estimating these bacteria.
Mono-acetyl: long red needles from EtOH. M.p. 222°.
Et ether: dark red needles from EtOH. M.p. 215°.

Nietzki, Dietze, Mäkler, *Ber.*, 1889, **22**, 3020.
Twigg, *Nature*, 1945, **155**, 401.

Reserpin.
Alkaloid of *Rauwolfia serpentina*, Benth. M.p. 262–3°. $[\alpha]_D^{23}$ −117–18° in $CHCl_3$. Sedative.

Müller, Schlittler, Bein, *Experientia*, 1952, **8**, 338.

Resobutyrophenone (2 : 4-*Dihydroxybutyrophenone, 4-butyrylresorcinol*)



$C_{10}H_{12}O_3$ MW, 180

Large needles from $CHCl_3$–C_6H_6. M.p. 68–70°. Sol. alkalis, EtOH, Et_2O, C_6H_6, AcOH. Spar. sol. H_2O, CCl_4. Antiseptic.

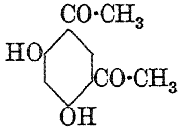
Oxime: pale yellow needles. M.p. 188–9°.

Johnson, Lane, *J. Am. Chem. Soc.*, 1921, **43**, 348.

Gnagy, *J. Am. Chem. Soc.*, 1923, **45**, 805.

Brewster, Harris, *J. Am. Chem. Soc.*, 1930, **52**, 4866.

Resodiacetophenone (4 : 6-*Diacetoresorcinol*)



$C_{10}H_{10}O_4$ MW, 194

Needles from EtOH. M.p. 185°. Sol. hot EtOH, hot C_6H_6, warm Et_2O, warm AcOH.

Diacetyl: m.p. 120°.

Dibenzoyl: m.p. 118°.

Dioxime: m.p. 242°.

Phenylhydrazone: m.p. 233°. *Monoacetyl deriv.*: m.p. 191–2°. *Monobenzoyl deriv.*: m.p. 214–15°.

Diphenylhydrazone: m.p. 291°.

Mono-Me ether: $C_{11}H_{12}O_4$. MW, 208. M.p. 121·5°. *Diphenylhydrazone*: m.p. 245–6°, decomp.

Di-Me ether: $C_{12}H_{14}O_4$. MW, 222. M.p. 171·5°.

Mono-Et ether: $C_{12}H_{14}O_4$. MW, 222. M.p. 109°.

Di-Et ether: $C_{14}H_{18}O_4$. MW, 250. M.p. 156°.

Me-Et ether: $C_{13}H_{16}O_4$. MW, 236. M.p. 152°.

Monopropyl ether: $C_{13}H_{16}O_4$. MW, 236. M.p. 94–5°.

Me-Propyl ether: $C_{14}H_{18}O_4$. MW, 250. M.p. 125·5°.

Et-Propyl ether: $C_{15}H_{20}O_4$. MW, 264. M.p. 95°.

Dipropyl ether: $C_{16}H_{22}O_4$. MW, 278. M.p. 86°.

Monoisopropyl ether: m.p. 97°.

Et-Isopropyl ether: m.p. 101·5°.

Propyl-Isopropyl ether: m.p. 78°.

Di-isopropyl ether: m.p. 126·5°.

Monobutyl ether: $C_{14}H_{18}O_4$. MW, 250. M.p. 63°.

Me-Butyl ether: $C_{15}H_{20}O_4$. MW, 264. M.p. 117°.

Et-Butyl ether: $C_{16}H_{22}O_4$. MW, 278. M.p. 104°.

Propyl-Butyl ether: $C_{17}H_{24}O_4$. MW, 292. M.p. 61·5°.

Isopropyl-Butyl ether: m.p. 76°.

Dibutyl ether: $C_{18}H_{26}O_4$. MW, 306. M.p. 92·5°.

Mono-isobutyl ether: m.p. 88°.

Me-Isobutyl ether: m.p. 102·5°.

Et-Isobutyl ether: m.p. 72°.

Propyl-Isobutyl ether: m.p. 64°.

Isopropyl-Isobutyl ether: m.p. 80°.

Butyl-Isobutyl ether: m.p. 78°.

Di-isobutyl ether: m.p. 99°.

Baker, *J. Chem. Soc.*, 1934, 71 (*Bibl.*); *ibid.*, 1684.

Israelstam, *Chem. Abstracts*, 1945, **39**, 2065

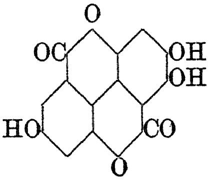
The name Resodiacetophenone has also been given to 2 : 4-diacetoresorcinol. M.p. 95–6° (92°).

Desai, Ekhlas, *Chem. Abstracts*, 1939, **33**, 2119.

Desai, Radha, *Chem. Abstracts*, 1940, **34**, 7874.

Israelstam, *Chem. Abstracts*, 1945, **39**, 2065.

Resoflavin (3 : 4 : 6 : 4′ : 6′-*Pentahydroxydiphenic acid dilactone*)



$C_{14}H_6O_7$ MW, 286

Yellow cryst. from EtOH. Decomp. about 380°. Zn dist. ⟶ fluorene.

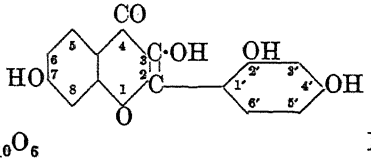
Triacetyl: m.p. 275–9°.

Tri-Me ether: $C_{17}H_{12}O_7$. MW, 328. M.p. 286–8°.

Badische, D.R.P., 85,390.

Herzig, Tscherne, *Ann.*, 1907, **351**, 30.

Resomorin (3 : 7 : 2′ : 4′-*Tetrahydroxyflavone, 7 : 2′ : 4′-trihydroxyflavonol*)



$C_{15}H_{10}O_6$ MW, 286

Cryst. Very sol. EtOH.

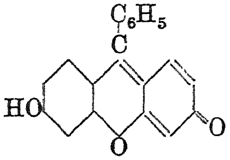
7 : 2′ : 4′-*Tri-Me ether*: $C_{18}H_{16}O_6$. MW, 328. Cryst. from C_6H_6 or EtOH. M.p. 205°. Conc. H_2SO_4 ⟶ yellowish sol. with intense bluish-

green fluor. *Acetyl*: prisms from EtOH.Aq. M.p. 189–91°.

Tetra-acetyl: needles from EtOH.Aq. M.p. 129–30°.

v. Kostanecki, Lampe, Triulzi, *Ber.*, 1906, **39**, 94.

Resorcin-benzein (*Resorcinol-benzein*)



$C_{19}H_{12}O_3$ MW, 288

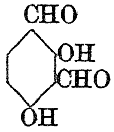
Red needles from $PhNO_2$. M.p. 330–1°. Sol. EtOH, Me_2CO, $PhNO_2$, aniline, Py. Spar. sol. Et_2O, $CHCl_3$. Sols. fluoresce.

6-*Acetyl*: m.p. 197°.

6-*Me ether*: $C_{20}H_{14}O_3$. MW, 302. Red needles from Et_2O. M.p. 206° (204°).

Liebig, *J. prakt. Chem.*, 1912, **85**, 244.
Gomberg, West, *J. Am. Chem. Soc.*, 1912, **34**, 1568.
Pope, Howard, *J. Chem. Soc.*, 1910, **97**, 1026.
Moir, *Chem. Abstracts*, 1923, **17**, 2283.

Resorcindialdehyde (2 : 4-*Di-hydroxyiso-phthaldehyde, resorcendialdehyde*)



$C_8H_6O_4$ MW, 166

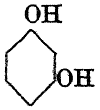
Needles from hot H_2O. M.p. 127°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Sublimes.

Dioxime: m.p. 209°.

Me ether: (i) Needles from hot H_2O. M.p. 179°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, AcOH. Insol. ligroin. (ii) Needles from hot H_2O. M.p. 88–9°. Sol. EtOH, Et_2O, C_6H_6, ligroin. Spar. sol. H_2O. Both forms volatile in steam.

Tiemann, Lewy, *Ber.*, 1877, **10**, 2211.
Tiemann, Parrisius, *Ber.*, 1880, **13**, 2369.
Baker *et. al.*, *J. Chem. Soc.*, 1932, 2876.

Resorcinol (1 : 3-*Dihydroxybenzene*)



$C_6H_6O_2$ MW, 110

Needles from C_6H_6. Plates from H_2O. (i) M.p. 110°, stable. (ii) M.p. 108–108·5°, labile. B.p. 273° (276·5°/759·7 mm.), 178°/16 mm. Sol. H_2O, EtOH, Et_2O. Mod. sol. $CHCl_3$, CS_2. Spar. sol. C_6H_6. $FeCl_3 \longrightarrow$ violet col. Heat of comb. C_v 683·1 Cal. $k = 3{\cdot}6 \times 10^{-10}$ at 18°. Reduces Tollen's and Fehling's. Sweet taste.

Mono-acetyl: curesol. B.p. 283°.

Diacetyl: b.p. 278°, 273°/708 mm.

Dichloroacetyl: m.p. 71·5–72° (76°). B.p. 191–2°/15 mm.

Di-iodoacetyl: m.p. 59–61°.

Di-1-bromopropionyl: m.p. 66°. B.p. 217–20°/10 mm.

Di-1-bromobutyryl: b.p. 225–7°/19 mm.

Di-1-bromoisobutyryl: b.p. 227–8°/20 mm.

Di-1-bromoisovaleryl: b.p. 222–8°/15 mm.

Penta-acetyl-β-glucoside: m.p. 118–19°. $[\alpha]_D^{15}$ — 40·1° in C_6H_6.

Monobenzoyl: m.p. 135–6° (133°).

Dibenzoyl: m.p. 117°.

Mono-salicyloyl: m.p. 141° (139°).

Disalicyloyl: m.p. 111°.

Di-p-nitrobenzoyl: m.p. 182°.

Dicinnamoyl: m.p. 119·5–120°.

Monohippuryl: *see* Hippurylresorcinol.

Di-benzenesulphonyl: m.p. 69–70°.

Di-p-toluenesulphonyl: m.p. 80–1°.

Di-d-camphor-β-sulphonyl: m.p. 129–30°.

Picrate: m.p. 89–90°.

Mono-Me ether: *m*-hydroxyanisole, *m*-methoxyphenol. $C_7H_8O_2$. MW, 124. B.p. 244°, 144°/25 mm., 102°/5 mm. *Acetyl*: b.p. 254–6°.

Di-Me ether: 1 : 3-dimethoxybenzene. $C_8H_{10}O_2$. MW, 138. M.p. — 52°. B.p. 216·5–217·7°. D_4^t 1·0705.

Mono-Et ether: *m*-hydroxyphenetole, *m*-ethoxyphenol. $C_8H_{10}O_2$. MW, 138. B.p. 246–7°, 117°/5·5 mm.

Di-Et ether: 1 : 3-diethoxybenzene. $C_{10}H_{14}O_2$ MW, 166. M.p. 12·4°. B.p. 234·4–235·2°/756 mm.

Me-Et ether: $C_9H_{12}O_2$. MW, 152. B.p. 216°.

Mono-propyl ether: $C_9H_{12}O_2$. MW, 152. B.p. 120°/5 mm.

Me-Propyl ether: $C_{10}H_{14}O_2$. MW, 166. B.p. 226°.

Dipropyl ether: $C_{12}H_{18}O_2$. MW, 194. B.p. 251°.

Monobutyl ether: $C_{10}H_{14}O_2$. MW, 166. B.p. 130°/5 mm.

Me-Isobutyl ether: $C_{11}H_{16}O_2$. MW, 180. B.p. 234°.

Monoamyl ether: $C_{11}H_{16}O_2$. MW, 180. B.p. 140°/5 mm.

Mono-isoamyl ether: b.p. 138°/5 mm.

Di-isoamyl ether: $C_{16}H_{26}O_2$. MW, 250. M.p. 47°.

Monohexyl ether: $C_{12}H_{18}O_2$. MW, 194. B.p. 145°/5 mm.

Monoheptyl ether: $C_{13}H_{20}O_2$. MW, 208. B.p. 160°/5 mm.

Mono-octyl ether: $C_{14}H_{22}O_2$. MW, 222. B.p. 170°/5 mm.

Mono-nonyl ether: $C_{15}H_{24}O_2$. MW, 236. B.p. 171°/4·5 mm.

Di-trichlorovinyl ether: $C_{10}H_4O_2Cl_6$. MW, 369. M.p. 53–4°.

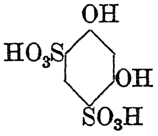
Phenyl ether: *see* 3-Hydroxydiphenyl Ether.
Diphenyl ether: $C_{18}H_{14}O_2$. MW, 262. M.p. 61·5°.
Di-2:4-dinitrophenyl ether: $C_{18}H_{10}O_{10}N_4$. MW, 442. M.p. 184°.
Monobenzyl ether: $C_{13}H_{12}O_2$. MW, 200. M.p. 69·2°. B.p. 200°/5 mm.
Mono-p-chlorobenzyl ether: $C_{13}H_{11}O_2Cl$. MW, 234·5. M.p. 76°. B.p. 235°/13 mm.
Dibenzyl ether: $C_{20}H_{18}O_2$. MW, 290. M.p. 76°.
Mono-phenylethyl ether: $C_{14}H_{14}O_2$. MW, 214. M.p. 44°. B.p. 202°/6 mm.
Mono-phenylpropyl ether: $C_{15}H_{16}O_2$. MW, 228. B.p. 220°/5·5 mm.
Mono-cyclohexyl ether: $C_{12}H_{16}O_2$. MW, 192. B.p. 160°/6 mm.
Me-α-phenylvinyl ether: $C_{15}H_{14}O_2$. MW, 226. B.p. 199–200°/16 mm.

Knoll, D.R.P., 281,099, (*Chem. Zentr.*, 1915, I, 179).
Phillips, Gibbs, *Ind. Eng. Chem.*, 1920, **12**, 857.
Carr, Dahlen, U.S.P., 1,999,955, (*Chem. Abstracts*, 1935, **29**, 4029).
Gallay, U.S.P., 1,956,570, (*Chem. Abstracts*, 1934, **28**, 4073).
Hayashi, *Chem. Abstracts*, 1934, **28**, 1679.
Ruth, *Chem. Abstracts*, 1933, **27**, 5316.
Klarmann, Gatyas, Shternov, *J. Am. Chem. Soc.*, 1931, **35**, 3397.
Fabre, *Ann. chim.*, 1922, **18**, 49.
Dey, *J. Indian Chem. Soc.*, 1935, **12**, 685.

Resorcinol-carboxylic Acid.
See Resorcylic Acid.

Resorcinol-dicarboxylic Acid.
See Dihydroxyisophthalic Acid *and* 2:6-Dihydroxyterephthalic Acid.

Resorcinol-4:6-disulphonic Acid

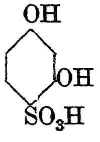


$C_6H_6O_8S_2$ MW, 270

Needles + $2H_2O$. Decomp. at 100°. Sol. H_2O, EtOH. Insol. Et_2O.
Dichloride: $C_6H_4O_6Cl_2S_2$. MW, 307. M.p. 178–9°. *Di-Me ether*: $C_8H_8O_6Cl_2S_2$. MW, 335. Yellow prisms from AcOEt. M.p. 175–8°. Sol. Et_2O, $CHCl_3$, AcOH, CS_2.
Diamide: $C_6H_8O_6N_2S_2$. MW, 268. Does not melt below 300°.
Dianilide: m.p. 262°.

Blumenstock, *Monatsh.*, 1925, **46**, 499.

Resorcinol-4-sulphonic Acid



$C_6H_6O_5S$ MW, 190

K salt: prisms + $2H_2O$ from H_2O.
Ba salt: hygroscopic cryst.

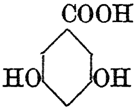
Darzens, Dubois, *Bull. soc. chim.*, 1892, 7, 713.

Resorcitol.
See Hexahydroresorcinol.

Resorcylaldehyde.
See Resorcylic Aldehyde.

β-Resorcylcyclohexane.
See 4-Hexahydrobenzoylresorcinol.

α-Resorcylic Acid (3:5-*Dihydroxybenzoic acid, resorcinol-5-carboxylic acid*)

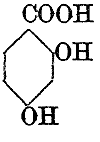


$C_7H_6O_4$ MW, 154

Cryst. + $1\frac{1}{2}H_2O$ from H_2O. M.p. 232–3° (237–40°) anhyd. Sol. EtOH, Et_2O, hot H_2O. $k = 9{\cdot}1 \times 10^{-5}$ at 25°. Oleum or hot conc. $H_2SO_4 \longrightarrow$ 1:2:4:5:6:8-hexahydroxy-anthraquinone. $FeCl_3 \longrightarrow$ deep port wine col.
Diacetyl: m.p. 156–7° (161–2°).
3:5-*Dibenzoyl*: needles from AcOH. M.p. 227°.
Dicarbomethoxyl: m.p. 161–4°.
Me ester: $C_8H_8O_4$. MW, 168. Leaflets. M.p. 163–5°. 3:5-*Dibenzoyl*: m.p. 121–2°. *Mono-Me ether*: $C_9H_{10}O_4$. MW, 182. B.p. 375° decomp. *Di-Me ether*: $C_{10}H_{12}O_4$. MW, 196. M.p. 42–4°. B.p. 298°, 157°/125 mm.
Et ester: m.p. 128·5°. *Hemihydrate*: m.p. *ca.* 80°.
Propyl ester: b.p. 215–17°/3 mm. *Monohydrate*: m.p. 67–8°.
Butyl ester: m.p. 62·5–63·5°. B.p. 209–10°/2 mm. *Hemihydrate*: m.p. 39–40°.
Amyl ester: b.p. 225–7°/4 mm.
Di-Me ether: *see* 3:5-Dimethoxybenzoic Acid.
Di-Et ether: $C_{11}H_{14}O_4$. MW, 210. M.p. 87–8°. *Et ester*: $C_{13}H_{18}O_4$. MW, 238. M.p. 19–20°. B.p. 212°/50 mm.

Zollinger, Roehling, U.S.P., 1,321,271, (*Chem. Abstracts*, 1920, **14**, 186).
Boehringer, D.R.P., 286,266, (*Chem. Zentr.*, 1915, II, 566).
Barth, Senhofer, *Ann.*, 1871, **159**, 222.
Suter, Weston, *J. Am. Chem. Soc.*, 1939, **61**, 531.
Birkinshaw, Bracken, *J. Chem. Soc.*, 1942, 368.

β-Resorcylic Acid (2:4-*Dihydroxybenozic acid, resorcinol-4-carboxylic acid*)



$C_7H_6O_4$ MW, 154

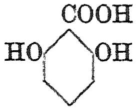
Cryst. from H_2O. M.p. 218–19° (213° rapid heat., 204–6°, 197° decomp.). Sol. EtOH, Et_2O, hot H_2O. $k = 5{\cdot}16 \times 10^{-4}$ at 25°. Heat of comb. C_p 676·9 Cal., C_v 677·2 Cal. Heat ⟶ resorcinol. $FeCl_3$ ⟶ red col. NaOCl ⟶ violet col.

2-*Acetyl*: m.p. 167–8° decomp. (rapid heat.).
4-*Acetyl*: m.p. 152–3°.
Diacetyl: m.p. 136–8°.
2-*Acetyl-4-benzoyl*: m.p. 148–9°.
Di-carbomethoxyl: m.p. 159°. *Chloride*: m.p. 86–7°.
2-*Benzoyl*: m.p. 160–1°. *Carbomethoxyl*: m.p. 148–9°.
4-*Benzoyl*: m.p. 193–4°.
Dibenzoyl: needles from AcOH. M.p. 152°.
Me ester: $C_8H_8O_4$. MW, 168. M.p. 118–19°. *Monohydrate*: m.p. 78–80°.
p-*Nitrobenzyl ester*: $C_{14}H_{11}O_6N$. MW, 289. M.p. 189°.
2-*Me ether*: $C_8H_8O_4$. MW, 168. Leaflets from H_2O. Decomp. at 187–9° (rapid heat.).
4-*Me ether*: 2-hydroxyanisic acid, 4-methoxysalicylic acid. M.p. 157° (about 161°). *Acetyl*: m.p. 145–7°. *Me ester*: $C_9H_{10}O_4$. MW, 182. Primula camphor. Occurs in root of *Primula veris.** Plates from ligroin. M.p. 49°. *Et ester*: $C_{10}H_{12}O_4$. MW, 196. B.p. 272–4°.
Di-Me ether: *see* 2 : 4-Dimethoxybenzoic Acid.
2-*Et ether*: $C_9H_{10}O_4$. MW, 182. Needles from H_2O. M.p. 154°. Sol. EtOH, Et_2O, C_6H_6, hot H_2O.
4-*Et ether*: *Me ester*: $C_{10}H_{12}O_4$. MW, 196. Plates from pet. ether. M.p. 77–9°. *Et ester*: $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH. M.p. 53–4° (45°).
Di-Et ether: $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH.Aq. M.p. 99°. *Me ester*: $C_{12}H_{16}O_4$. MW, 224. Cubes from EtOH.Aq. M.p. 51–4°.
Chloride: $C_7H_5O_3Cl$. MW, 172·5. F.p. − 20°. B.p. 170°/12 mm.
Amide: $C_7H_7O_3N$. MW, 153. M.p. 228–9°.
Diethylamide: m.p. 139°.
Nitrile: 4-cyanoresorcinol. $C_7H_5O_2N$. MW, 135. M.p. 175°. Sol. H_2O, EtOH. *Diacetyl*: m.p. 72°.
Anilide: m.p. 126–7°.

Shoesmith, Haldane, *J. Chem. Soc.*, 1924, **125**, 113.
Miksic, *J. prakt. Chem.*, 1928, **119**, 218.
Clibbens, Nierenstein, *J. Chem. Soc.*, 1915, **107**, 1493; *Organic Syntheses*, 1930, X, 94.
Robinson, Shah, *J. Chem. Soc.*, 1934, 1496.
Mauthner, *J. prakt. Chem.*, 1930, **124**, 319.
Bergmann, Dangschat, *Ber.*, 1919, **52**, 371.
King, McWhirter, Barton, *J. Am. Chem. Soc.*, 1945, **67**, 2089.

**Note.*—*Primula veris* is the synonym of three different *Primula* species. Since the authority is not given in the original paper it is impossible to assign the correct Index Kewensis name to this plant.

γ-Resorcylic Acid (2 : 6-*Dihydroxybenzoic acid, resorcinol-2-carboxylic acid*)



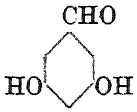
$C_7H_6O_4$ MW, 154

Cryst. + $1H_2O$ from H_2O. M.p. varies from 150 to 170° according to rate of heating ⟶ resorcinol. Sol. EtOH, Et_2O, hot H_2O. $k = 6 \times 10^{-2}$ at 25°. Reduces warm Fehling's but not Tollen's. $FeCl_3$ ⟶ violet to blue col.

Me ester: $C_8H_8O_4$. MW, 168. M.p. 67–8°.
Me ether: $C_8H_8O_4$. MW, 168. Cryst. from $CHCl_3$–C_6H_6. M.p. 135°.
Di-Me ether: *see* 2 : 6-Dimethoxybenzoic Acid.
Di-Et ether: $C_{11}H_{14}O_4$. MW, 210. M.p. 122°.

Brunner, *Ann.*, 1907, **351**, 320.
Mauthner, *J. prakt. Chem.*, 1930, **124**, 319.
Jenkins, *Nature*, 1943, **151**, 561.

α-Resorcylic Aldehyde (3 : 5-*Dihydroxybenzaldehyde*)



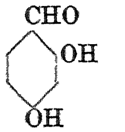
$C_7H_6O_3$ MW, 138

Needles from ligroin. M.p. 156–7°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6, ligroin.

5-*Me ether*: sublimes at 110–120°. M.p. 130–1°.
Semicarbazone: needles. M.p. 223–4° decomp.
p-*Nitrophenylhydrazone*: chars without melting at 280°.

Mauthner, *J. prakt. Chem.*, 1921, **101**, 94.
Späth, Kromp, *Ber.*, 1941, **74**, 1424.

β-Resorcylic Aldehyde (2 : 4-*Dihydroxybenzaldehyde*)



$C_7H_6O_3$ MW, 138

Needles from Et_2O–ligroin. M.p. 201–2°. B.p. 220–8°/22 mm. Sol. H_2O, EtOH, Et_2O, $CHCl_3$, AcOH. Spar. sol. C_6H_6. Gives deep brown col. with $FeCl_3$, yellow ppt. with Schiff's reagent.

Diacetyl: m.p. 69°.
Dibenzoyl: m.p. 98°.

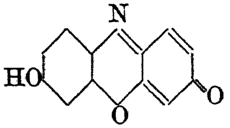
Di-carbomethoxyl: m.p. 72°. *Phenylhydrazone*: m.p. 138°. *Semicarbazone*: m.p. 185°.
Oxime: m.p. 192°.
Semicarbazone: decomp. at 260°.
Phenylhydrazone: m.p. 156–60° decomp.
3-*Nitrophenylhydrazone*: m.p. 205–6°.
4-*Nitrophenylhydrazone*: m.p. 285° decomp.
unsym.-*Me*-p-*nitrophenylhydrazone*: m.p. 265°.
2 : 4-*Dinitrophenylhydrazone*: m.p. 286° decomp. (266–7°).
3 : 4-*Dichloro*-6-*nitrophenylhydrazone*: m.p. 251°.
Anil: m.p. 125–6°.
2-*Me ether*: $C_8H_8O_3$. MW, 152. M.p. 153°.
4-*Acetyl*: m.p. 86°.
4-*Me ether*: 2-hydroxyanisaldehyde, 4-methoxysalicylaldehyde. M.p. 40–2°.
Di-Me ether: 2-methoxyanisaldehyde. $C_9H_{10}O_3$. MW, 166. M.p. 68–9° (71°). B.p. 165°/10 mm.
4-*Et ether*: 4-ethoxysalicylaldehyde. $C_9H_{10}O_3$. MW, 166. M.p. 35°.
Di-Et ether: $C_{11}H_{14}O_3$. MW, 194. M.p. 71–2°.

Karrer, *Helv. Chim. Acta*, 1919, **2**, 89.
Weil, Traun, Marcel, *Ber.*, 1922, **55**, 2665.
Mitter, Suha, *J. Indian Chem. Soc.*, 1934, **11**, 257.
Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2798.
Shoesmith, Haldane, *J. Chem. Soc.*, 1923, **123**, 2704.
Scott, Burns, *J. Am. Chem. Soc.*, 1940, **62**, 3532.
Scott, McCall, *J. Am. Chem. Soc.*, 1945, **67**, 1767.

γ-Resorcylic Aldehyde (2 : 6-*Dihydroxybenzaldehyde*).
M.p. 155–6°. $FeCl_3$ ⟶ dark brown col.
Oxime: m.p. 167°.
Semicarbazone: m.p. 245°.
2 : 4-*Dinitrophenylhydrazone*: yellow cryst. M.p. 288–91°.
2-*Me ether*: m.p. 75°. Alc.$FeCl_3$ ⟶ reddish-violet col. *Semicarbazone*: m.p. 250°.

Shah, Laiwalla, *Chem. Zentr.*, 1937, I, 1679; *J. Chem. Soc.*, 1938, 1828.

Resorufin (*Azoresorufin*, 9-*hydroxy*-3-*isophenoxazone*, *diazoresorufine*)



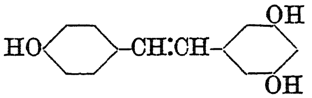
$C_{12}H_7O_3N$ MW, 213

Brown needles from aniline. Sol. alkalis. Spar. sol. EtOH. Insol. H_2O, Et_2O.
Acetyl deriv.: m.p. 221°.

Et ether: $C_{14}H_{11}O_3N$. MW, 241. Orange-red needles from EtOH. M.p. 225°.

Eichler, *J. prakt. Chem.*, 1934, **139**, 113.
Nietzki, Dietze, Mäckler, *Ber.*, 1889, **22**, 3020.

Resveratrol (3 : 5 : 4′-*Trihydroxystilbene*)

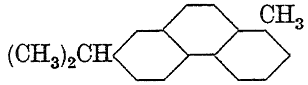


$C_{14}H_{12}O_3$ MW, 228

Obtained from roots of white hellebore (*Veratrum grandiflorum*, Loes. fil.). Cryst. from EtOH.Aq. M.p. 261° decomp. Absorption maxima at 3610, 3350, 3020, 2900 and 2480 Å. $FeCl_3$ ⟶ dark green col.
Triacetyl: needles from EtOH. M.p. 114–16°.
Tri-Me ether: cryst. from MeOH. M.p. 56–7°. B.p. 140–60°/0·01 mm.

Takaoka, *Chem. Abstracts*, 1940, **34**, 7887; 1941, **35**, 1398.
Späth, Kromp, *Ber.*. 1941, **74**, 867.

Retene (1-*Methyl*-7-*isopropylphenanthrene*)

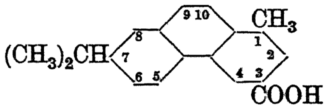


$C_{18}H_{18}$ MW, 234

Plates from EtOH. M.p. 100·5–101° (98–9°). B.p. 390°, 158–65°/0·2 mm. Sol. C_6H_6, ligroin, CS_2, hot EtOH, hot AcOH. Heat of comb. C_p 2326·1 Cal., C_v 2323·6 Cal.
Picrate: m.p. 124–5°.
Styphnate: m.p. 141–2°.
$C_{18}H_{18},C_6H_3(NO_3)_3$-1 : 3 : 5 : m.p. 139–40°.

Cheung, *Chem. Abstracts*, 1929, **23**, 4464, (*Bibl.*).
Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1791.
Nagel, Körnchen, *Chem. Abstracts*, 1932, **26**, 1808.
Bardhan, Sengupta, *J. Chem. Soc.*, 1932, 2798.
Henke, Etzel, U.S.P., 1,881,565, (*Chem. Abstracts*, 1933, **27**, 516).
Nyman, *Chem. Zentr.*, 1936, I, 2348.
Ruzicka, Waldman, *Helv. Chim. Acta*, 1933, **16**, 847.
Hosking, McFadyen, *J. Soc. Chem. Ind.*, 1934, **53**, 195T.
Darzens, Lévy, *Chem. Zentr.*, 1937, I, 592.
Keimatsu, Ishiguro, *Chem. Abstracts*, 1935, **29**, 7323, (*Refs.*).

Retene-3-carboxylic Acid (1-*Methyl*-7-*isopropylphenanthrene*-3-*carboxylic acid*)



$C_{19}H_{18}O_2$ MW, 278

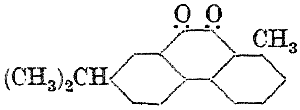
M.p. 231–4°.
Amide: m.p. 229–32°.
Nitrile: needles. M.p. 155·5–156°.

Elderfield, Dodd, Gensler, *J. Org. Chem.*, 1947, **12**, 393.

Retene-6-carboxylic Acid (*1-Methyl-7-iso-propylphenanthrene-6-carboxylic acid*).
Needles from C_6H_6. M.p. 241–3° (238–238·5°).

Nyman, *Chem. Zentr.*, 1936, I, 2349.
Adelson, Bogert, *J. Am. Chem. Soc.*, 1936, **58**, 654.

Retenequinone (*1-Methyl-7-isopropylphen-anthraquinone*)

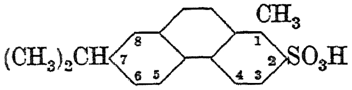


$C_{18}H_{16}O_2$ MW, 264

Orange needles from $CHCl_3$–EtOH. M.p. 197–9°. Sol. EtOH, Et_2O. Insol. AcOH, C_6H_6, $CHCl_3$. Conc. H_2SO_4 ⟶ olive green col.
Oxime: golden needles from EtOH. M.p. 128·5°.

Bamberger, Hooker, *Ann.*, 1885, **229**, 122.
Kreps, Day, *J. Org. Chem.*, 1941, **6**, 140.
Steck, Day, *J. Am. Chem. Soc.*, 1943, **65**, 452.

Retene-2(?)-sulphonic Acid



$C_{18}H_{18}O_3S$ MW, 314

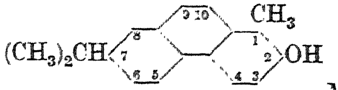
M.p. 188–9°.
Me ester: $C_{19}H_{20}O_3S$. MW, 328. M.p. 164–6°.
Et ester: $C_{20}H_{20}O_3S$. MW, 342. M.p. 137·5–138·5°.
Chloride: $C_{18}H_{17}O_2ClS$. MW, 332·5. Yellow scales from Et_2O. M.p. 135–6°.

Komppa, Fogelberg, *J. Am. Chem. Soc.*, 1932, **54**, 2907.

Retene-6-sulphonic Acid.
M.p. 121–3°.
Me ester: m.p. 117–19°.
Et ester: m.p. 114–15°.
Chloride: yellow needles from C_6H_6. M.p. 146–147·5°.
Amide: $C_{18}H_{19}O_2NS$. MW, 313. M.p. 206–207·5°.

Komppa, Fogelberg, *J. Am. Chem. Soc.*, 1932, **54**, 2907.
Hasselstrom, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 1580.

2-Retenol (*2-Hydroxyretene*)



$C_{18}H_{18}O$ MW, 250

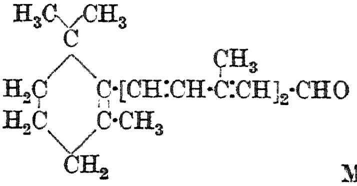
M.p. 153°. Sol. Et_2O.

Kuo, *J. Chinese Chem. Soc.*, 1945, **12**, 15.

6-Retenol (*6-Hydroxyretene*).
Yellow scales from xylene. M.p. 161°.
Acetyl: m.p. 134–5°.
p-*Toluenesulphonyl*: cryst. from EtOH. M.p. 110·5–111°.
Picrate: deep red cryst. from EtOH. M.p. 152–152·5°.

Adelson, Bogert, *J. Am. Chem. Soc.*, 1936, **58**, 653.
Karrman, *Svensk Kem. Tid.*, 1945, **57**, 14, (*Chem. Abstracts*, 1946, **40**, 4371).

Retinene₁ (*Vitamin A_1 aldehyde*)

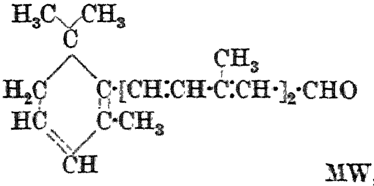


$C_{20}H_{28}O$ MW, 284

Together with protein forms part of Rhodopsin (Visual Purple). M.p. 64–5° (61–2°). $SbCl_3$ in $CHCl_3$ ⟶ deep blue col. Red. with $LiAlH_4$ ⟶ Vitamin A_1.
Semicarbazone: yellow cryst. from $CHCl_3$–Et_2O. M.p. 188–90° decomp.
Oxime: orange-yellow needles from MeOH. M.p. 134–6°.
2 : 4-*Dinitrophenylhydrazone*: deep purple needles from AcOEt. M.p. 214–15°.

Heilbron, Johnson, Jones, *J. Chem. Soc.*, 1939, 1560.
Wendler, Rosenblum, Tishler, *J. Am. Chem. Soc.*, 1950, **72**, 234.
Johnson, *Annual Reports of the Chemical Society* (London), 1949, XLVI, 181.
Farrar, Hamlet, Henbest, Jones, *J. Chem. Soc.*, 1952, 2657.

Retinene₂ (*Vitamin A_2 aldehyde*)



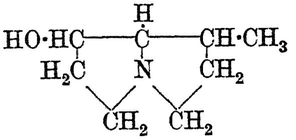
$C_{20}H_{26}O$ MW, 282

Together with protein forms part of Rhodopsin (Visual Purple). Orange-red prisms. M.p. 77–8°. Red. with $LiAlH_4$ ⟶ Vitamin A_2.
Oxime: dark yellow needles from MeOH. M.p. 141–3°.

2 : 4-*Dinitrophenylhydrazone* : violet-black needles from AcOEt. M.p. 197–9°.

Heilbron, Johnson, Jones, *J. Chem. Soc.*, 1939, 1560.
Morton, Salah, Stubbs, *Nature*, 1947, **159**, 744.
Wendler, Rosenblum, Tishler, *J. Am. Chem. Soc.*, 1950, **72**, 234.
Johnson, *Annual Reports of the Chemical Society*, (London), 1949, XLVI, 181.
Farrar, Hamlet, Henbest, Jones, *J. Chem. Soc.*, 1952, 2657.

Retronecanol



$C_8H_{15}ON$ MW, 141

Occurs in *Senecio longilobus* and *Crotalaria spectabilis*. Cryst. from pet. ether. M.p. 95–6°. $[\alpha]_D$ — 105°.

B,HCl : white prisms from $MeOH–Et_2O$. M.p. 210°.

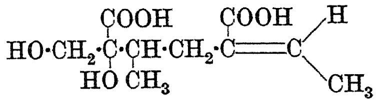
Methiodide : white plates from $MeOH–Et_2O$. M.p. 193°. $[\alpha]_D$ — 53° in MeOH.

Picrate : thin yellow plates from H_2O. M.p. 210°.

Picrolonate : yellow prisms from EtOH. M.p. 184–5°.

Adams, Govindachari, *J. Am. Chem. Soc.*, 1949, **71**, 1180.
Konovalova, Orekhov, *Bull. soc. chim.*, 1937, **4**, 1285.

Retronecic Acid (6 : 7-*Dihydroxy*-5-*methyl*-2-*heptene*-3 : 6-*dicarboxylic acid*)



$C_{10}H_{16}O_6$ MW, 232

M.p. 180–181·5°. $[\alpha]_D$ + 58°.

Warren *et al.*, *J. Chem. Soc.*, 1949, 1700, 1703.

Retronecine (*Senecifolinene* ?)

$C_8H_{13}O_2N$ MW, 155

M.p. 121–2°. Sol. H_2O, EtOH. Mod. sol. Me_2CO, $CHCl_3$. Spar. sol. Et_2O. $[\alpha]_D$ + 50–2° in EtOH. Alkaline. Forms no picrate.

B,HCl : m.p. 162–3° (161°). $[\alpha]_D^{15}$ — 16° in EtOH.

Aurichloride : m.p. 146°.

Diacetyl deriv. : *picrate*, m.p. 146°. *Methiodide* : m.p. 118–20°.

Barger, Seshadri, Watt, Yabuta, *J. Chem. Soc.*, 1935, 13.
Barger, Blackie, *J. Chem. Soc.*, 1936, 744.

Retrorsine

$C_{18}H_{25}O_6N$ MW, 351

Occurs in *Senecio barbellatus*, D.C. Leaflets from AcOEt. M.p. 214–15° (212°). Sol. EtOH, $CHCl_3$. Spar. sol. H_2O, Et_2O, AcOEt, Me_2CO. $[\alpha]_D^{18}$ — 17·6° in EtOH.

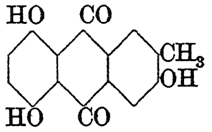
$B,HNO_3,\frac{1}{2}EtOH$: m.p. 145°.

Phenylurethane : m.p. 200–2°.

Methiodide : m.p. 260°.

Barger, Seshadri, Watt, Yabuta, *J. Chem. Soc.*, 1935, 11.

Rhababerone (*Isoemodin*, 3 : 5 : 8-*trihydroxy*-2-*methylanthraquinone*)

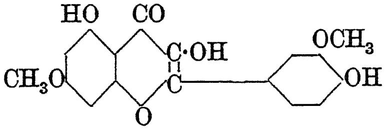


$C_{15}H_{10}O_5$ MW, 270

Occurs in rhubarb. Yellow leaflets from hot EtOH. M.p. 223–4° (212°). Sol. hot. EtOH, hot AcOH. Spar. sol. Et_2O. Insol. H_2O. $FeCl_3 \longrightarrow$ dark brown col. in EtOH. Sol. alkalis $\longrightarrow$ purple-red col.

Keimatsu, Hirano, *Chem. Abstracts*, 1931, **25**, 3647; *Chem. Zentr.*, 1931, I, 3348.

Rhamnazin (3 : 5 : 4′-*Trihydroxy*-7 : 3′-*dimethoxyflavone*, *quercetin* 7 : 3′-*dimethyl ether*)



$C_{17}H_{14}O_7$ MW, 330

Found in fruits of *Rhamnus infectoria*, Linn. Pale yellow needles from toluene. M.p. 214–15° (216–18°). Mod. sol. boiling AcOH, toluene. Spar. sol. EtOH. Alkalis $\longrightarrow$ orange-red sols. $FeCl_3 \longrightarrow$ olive-green col. Conc. $H_2SO_4 \longrightarrow$ yellow sol. with brilliant green fluor.

Triacetyl : needles from EtOH. M.p. 154–5°.

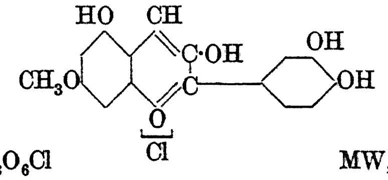
Tribenzoyl : needles from AcOH. M.p. 204–5°. Spar. sol. AcOH.

Perkin, Allison, *J. Chem. Soc.*, 1902, **81** 469.
Kuhn, Löw, Trischmann, *Ber.*, 1944, **77**, 217.
Rao, Seshadri, *J. Chem. Soc.*, 1946, 771.

α-Rhamnegin.

See Xanthorhamnin.

Rhamnetidin chloride

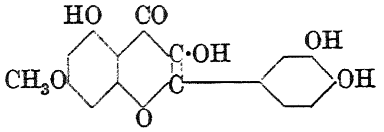


$C_{16}H_{13}O_6Cl$ MW, 336·5

Purplish-brown prisms + $2H_2O$. Orange-red by transmitted light. Green reflex. Sol. EtOH ⟶ bluish-red col. Sol. hot HCl ⟶ brownish-red col. Na_2CO_3 ⟶ blue col. $FeCl_3$ ⟶ blue col. in EtOH, violet in H_2O.

> Kondo, Segawa, *Chem. Abstracts*, 1932, **26**, 4333.
> Robertson, Robinson, *J. Chem. Soc.*, 1927, 2205.

Rhamnetin (*Quercetin 7-methyl ether, 3 : 5 : 3′ : 4′-tetrahydroxy-7-methoxyflavone, β-rhamnocitrin*)



$C_{16}H_{12}O_7$ MW, 316

Occurs in fruit of *Rhamnus cathartica*, Linn., and other species. Yellow needles from EtOH or phenol. M.p. 294–6°. Sol. Me_2CO, hot EtOH. Spar. sol. H_2O. $FeCl_3$ ⟶ brown col. Conc. H_2SO_4 ⟶ yellow col. with greenish-blue fluor. Reduces cold Tollen's and warm Fehling's.

Tetra-acetyl: m.p. 190–2° (186–8°).

Tetra-propionyl: m.p. 158–62°.

> Herzig, *Monatsh.*, 1888, **9**, 549.
> Liebermann, Hörmann, *Ann.*, 1879, **196**, 313.
> Krassowski, *Chem. Zentr.*, 1909, I, 772.
> Oesch, Perkin, *J. Chem. Soc.*, 1914, **105**, 2354.
> Kuhn, Löw, Trischmann, *Ber.*, 1944, **77**, 216.

Rhamninose

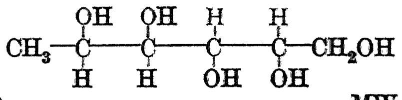
$C_{18}H_{32}O_{14}$ MW, 472

Obtained from xanthorhamnin present in Persian berries (*Rhamnus infectoria*). Cryst. M.p. 135–40° decomp. Sweet taste. $[\alpha]_D$ − 41·0° in H_2O, − 26·37° in 75% EtOH. Sol. H_2O, EtOH. Insol. Et_2O, Me_2CO, AcOEt. Reduces Fehling's. Hyd. by dil. acids ⟶ 2 mols. rhamnose + 1 mol. galactose. NaHg ⟶ rhamninitol. Br water ⟶ rhamnotrionic acid.

Octa-acetyl: cryst. M.p. 95°. $[\alpha]_D$ − 30·87° in EtOH, − 31·7° in AcOH.

> Ter Meulen, *Rec. trav. chim.*, 1923, **42**, 380.
> Tanret, Tanret, *Bull. soc. chim.*, 1899, **21**, 1065.

Rhamnitol



$C_6H_{14}O_5$ MW, 166

d-.

Prisms from Me_2CO. M.p. 123°. Sol. H_2O, EtOH. Spar. sol. $CHCl_3$. Insol. Et_2O. $[\alpha]_D^{20}$ − 12·4° in H_2O.

Dibenzylidene deriv.: m.p. 207°. $[\alpha]_D$ + 60·7° in $CHCl_3$.

l-.

M.p. 121°. $[\alpha]_D^{20}$ + 10·7° in H_2O.

Dibenzylidene deriv.: cryst. M.p. 203°. $[\alpha]_D$ − 55° in $CHCl_3$.

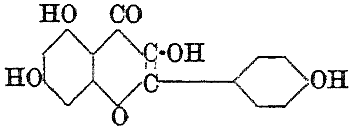
3 : 4-*Di-Me ether*: m.p. 105°. $[\alpha]_D^{24}$ − 25·5° in MeOH. n_D^{25} 1·4713 (superfused).

3 : 4 : 5-*Tri-Me ether*: b.p. 99–100°/0·1 mm. $[\alpha]_D^{29}$ − 14·8° in MeOH. n_D^{25} 1·4534.

1 : 3 : 4 : 5-*Tetra-Me ether*: b.p. 87°/0·25 mm. $[\alpha]_D^{29}$ − 8·1° in EtOH.

> Votoček, Valentin, Rac, *Chem. Abstracts*, 1931, **25**, 84.
> Fischer, Piloty, *Ber.*, 1890, **23**, 3103.
> de Bruyn, van Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151.
> Gätzi, Reichstein, *Helv. Chim. Acta*, 1938, **21**, 914.
> Tipson, Levene, *J. Biol. Chem.*, 1939, **130**, 235.
> Haskins, Hann, Hudson, *J. Am. Chem. Soc.*, 1946, **68**, 628.

Rhamnocitrin (*Kæmpferol methyl ether*)



One of the OH groups is methylated

$C_{16}H_{12}O_6$ MW, 300

Occurs in fruits of *Rhamnus cathartica*, Linn. Yellow needles. M.p. 221–2°. Mod. sol. Me_2CO, AcOEt. Spar. sol. hot EtOH. Insol. H_2O, Et_2O, C_6H_6. Conc. H_2SO_4 ⟶ green fluor. Reduces Fehling's.

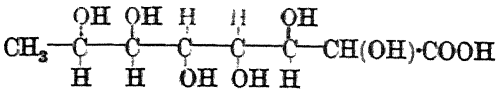
Triacetyl: m.p. 200–1°.

> Oesch, Perkin, *J. Chem. Soc.*, 1914, **105**, 2352.

β-Rhamnocitrin.

See Rhamnetin.

Rhamnoheptonic Acid



$C_8H_{16}O_8$ MW, 240

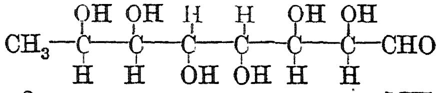
Free acid not isolated.

γ-Lactone: $C_8H_{14}O_7$. MW, 222. Needles from EtOH. M.p. 160°. $[\alpha]_D^{20}$ + 55·6° in H_2O. Sol. H_2O. Mod. sol. MeOH, EtOH. Insol. Et_2O.

Phenylhydrazide: needles from H_2O. M.p. about 215° decomp. Spar. sol. H_2O, EtOH.

Fischer, Piloty, *Ber.*, 1890, **23**, 3106.

Rhamnoheptose



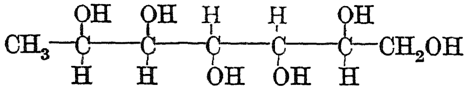
$C_8H_{16}O_7$ MW, 224

Syrup. Sol. H_2O, EtOH. Insol. Et_2O. $[\alpha]_D^{20}$ + 8·4° in H_2O.

Phenylosazone: yellow needles. M.p. 200° decomp.

Anderson, *J. Am. Chem. Soc.*, 1911, **33**, 1514.
Fischer, Piloty, *Ber.*, 1890, **23**, 3107.

α-Rhamnohexitol

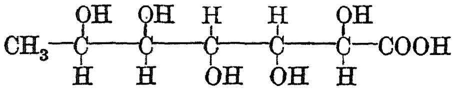


$C_7H_{16}O_6$ MW, 196

Prisms from EtOH. M.p. 173°. $[\alpha]_D$ + 14° in H_2O. Mod. sol. hot MeOH, hot EtOH.

Fischer, Piloty, *Ber.*, 1890, **23**, 3106.
Valentin, *Chem. Abstracts*, 1932, **26**, 1578.

α-Rhamnohexonic Acid



$C_7H_{14}O_7$ MW, 210

M.p. 171°. $[\alpha]_D^{20}$ + 21·5° in H_2O.

NH_4 salt: m.p. 151°.

Brucine salt: m.p. 120–3°.

Et ester: $C_9H_{18}O_7$. MW, 238. M.p. 165–6° decomp. $[\alpha]_D^{20}$ + 12·9° in H_2O.

Amide: $C_7H_{15}O_6N$. MW, 209. M.p. 177·5–178° decomp. (194°). $[\alpha]_D^{20}$ − 19·9° in H_2O (− 47·26°).

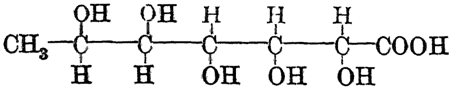
Nitrile: $C_7H_{13}O_5N$. MW, 191. M.p. 145°. $[\alpha]_D^{20}$ − 23·47°.

γ-Lactone: $C_7H_{12}O_6$. MW, 192. Cryst. from Me_2CO. M.p. 171–171·5° (174·5°). $[\alpha]_D^{20}$ + 87·3° in H_2O.

Phenylhydrazide: m.p. 205–6° decomp. $[\alpha]_D^{20}$ − 5·2° in H_2O.

Brackenbury, Upson, *J. Am. Chem. Soc.*, 1934, **56**, 2659.
Jackson, Hudson, *ibid.*, 2455.
Miksic, *Chem. Abstracts*, 1929, **23**, 2942.
Fischer, Morrell, *Ber.*, 1894, **27**, 386.

β-Rhamnohexonic Acid



$C_7H_{14}O_7$ MW, 210

Free acid not isolated.

Brucine salt: m.p. 114–18°.

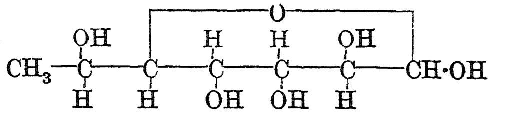
γ-Lactone: plates from Me_2CO. M.p. 134–8°. $[\alpha]_D^{20}$ + 43·34° in H_2O.

Phenylhydrazide: m.p. 170° decomp.

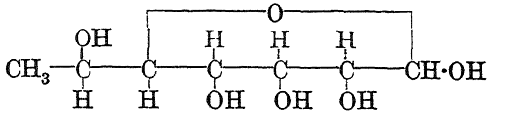
Fischer, Morrell, *Ber.*, 1894, **27**, 389.

Rhamnohexose

α-



β-



$C_7H_{14}O_6$ MW, 194

α-

l-.

Prisms or plates from hot MeOH. M.p. 180–1°. Sol. H_2O, hot MeOH. Spar. sol. EtOH. $[\alpha]_D^{20}$ − 80° ⟶ − 61·4° in H_2O. Sweet taste. NaHg ⟶ α-rhamnohexitol.

Phenylosazone: yellow needles. M.p. 200° decomp. Sol. hot EtOH. Insol. H_2O.

Benzylphenylhydrazone: m.p. 183–4°.

β-

l-.

Syrup.

Phenylosazone: identical with that of α-form.

Monobenzylidene deriv.: m.p. 233–4°. $[\alpha]_D$ + 50–8° in $CHCl_3$.

Votoček, Valentin, Leminger, *Chem. Abstracts*, 1931, **25**, 4527.
Votoček, Valentin, Rac, *ibid.*, 84.
Anderson, *J. Am. Chem. Soc.*, 1911, **33**, 1514.
Fischer, Morrell, *Ber.*, 1894, **27**, 391.

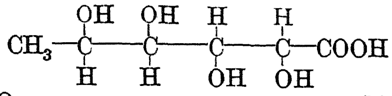
Rhamnol.

See β-Sitosterol.

Rhamnolutin.

See Kæmpferol.

Rhamnonic Acid



$C_6H_{12}O_6$ MW, 180

l-.

Obtained only in solution. $[\alpha]_D$ − 7·67 ⟶ 29·28° in H_2O.

Brucine salt: m.p. 120–6° (132°).

Quinine salt: m.p. 180–1°.

Nitrile: *tetra-acetyl deriv.*: m.p. 69–70°.

γ-Lactone: $C_6H_{10}O_5$. MW, 162. M.p. 149–51°. $[\alpha]_D^{20}$ − 39·2° in H_2O.

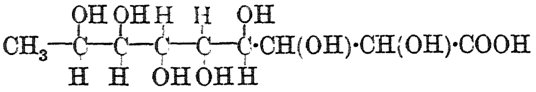
δ-Lactone: m.p. 178–82°. $[\alpha]_D$ + 98–101°.

Amide: $C_6H_{13}O_5N$. MW, 179. M.p. 134–134·5°. $[\alpha]_D^{20}$ + 27·7° in H_2O.

Phenylhydrazide: m.p. 195–6°. $[\alpha]_D^{20}$ + 17·2° in H_2O. 2 : 3 : 5-*Tri-Me ether*: m.p. 160°.

Brackenbury, Upson, *J. Am. Chem. Soc.*, 1933, **55**, 2514.
Isbell, Frush, *Chem. Abstracts*, 1934, **28**, 1667.
Rehorst, *Ann.*, 1933, **503**, 143.
Smith, *J. Chem. Soc.*, 1940, 1035.

Rhamno-octonic Acid



$C_9H_{18}O_9$ MW, 270

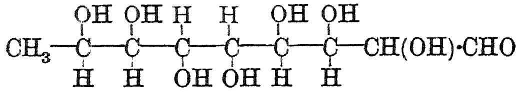
Free acid not isolated.

γ-Lactone: $C_9H_{16}O_8$. MW, 252. Needles from H_2O. M.p. 171–2°. $[\alpha]_D^{20}$ − 50·8° in H_2O. Sol. H_2O, EtOH. Spar. sol. Me_2CO. NaHg ⟶ rhamno-octose.

Phenylhydrazide: needles from H_2O. M.p. about 220° decomp. Spar. sol. hot H_2O.

Fischer, Piloty, *Ber.*, 1890, **23**, 3109, 3827.

Rhamno-octose



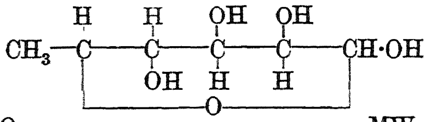
$C_9H_{18}O_8$ MW, 254

Syrup. Sol. H_2O.

Phenylosazone: m.p. 216°.

Fischer, Piloty, *Ber.*, 1890, **23**, 3110.

Rhamnose



$C_6H_{12}O_5$ MW, 164

d-.

Cryst. + H_2O. Sol. H_2O. Insol. Et_2O. $[\alpha]_D^{16\cdot5}$ − 8·25° in H_2O. Reduces warm Fehling's. NaHg ⟶ *d*-rhamnitol.

Phenylosazone: yellow prisms or needles from dil. EtOH. M.p. 191° (185°, 186–7°, 189–90°). $[\alpha]_D^{20}$ − 95·2° in Py. Sol. EtOH, Py. Identical with phenylosazone of *d*-isorhodeose and *d*-isorhamnose.

p-*Bromophenylosazone*: yellow cryst. from dil. EtOH. M.p. 225° (222–3°).

l-.

α-Form:

A constituent of many glycosides. Cryst. + H_2O from H_2O. M.p. 105° (93–4°). Sweet taste. $[\alpha]_D^{15}$ + 9·1° in H_2O ($[\alpha]_D^{20}$ − 7·7° ⟶ + 8·9° in H_2O.) Sol. H_2O, MeOH. Reduces warm Fehling's.

Oxime: plates. M.p. 127–8°. $[\alpha]_D^{20}$ + 13·70° in H_2O (final). Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O.

Semicarbazone: cryst. from dil. EtOH. M.p. 183°. $[\alpha]_D^{20}$ + 75° ⟶ + 57° in H_2O.

Phenylhydrazone: laminæ. M.p. 159°. $[\alpha]_D^{20}$ + 54·2° in H_2O, $[\alpha]_D$ + 27° in EtOH. Mod. sol. H_2O, EtOH. Insol. Et_2O.

Diphenylhydrazone: prisms. M.p. 134°. Sol. H_2O, EtOH. Insol. Et_2O.

p-*Nitrophenylhydrazone*: reddish-yellow cryst. M.p. 186° (191–2°). $[\alpha]_D$ + 21·4° (− 50·3° ⟶ − 8·5°) in EtOH–Py. Sol. EtOH.

Methylphenylhydrazone: cryst. M.p. 124°. $[\alpha]_D$ + 0·7° in MeOH. Sol. MeOH. Spar. sol. H_2O, EtOH.

Benzylphenylhydrazone: yellow cryst. M.p. 121°. $[\alpha]_D$ − 6·4° in MeOH.

β-*Naphthylhydrazone*: brown needles. M.p. 170°. $[\alpha]_D$ + 8·4° in MeOH, − 11·8° in AcOH. Sol. MeOH. Spar. sol. H_2O.

Phenylosazone: yellow needles. M.p. 222° (180°, 185°, 186–7°, 182°). $[\alpha]_D^{20}$ + 93·92° (94°) in Py. Sol. hot EtOH, AcOH, Me_2CO. Spar. sol. EtOH, C_6H_6. Insol. H_2O. Reduces boiling Fehling's.

p-*Nitrophenylosazone*: red needles from EtOH. M.p. 208° decomp. Spar. sol. EtOH.

4-d-*Glucoside*: *see* Scillabiose.

Methylglycoside: cryst. from AcOEt. M.p. 108–9° (109–10°). $[\alpha]_D^{20}$ − 62·5° (− 25·43°) in H_2O. *Tri-Me ether*: b.p. 112°/11 mm., 101°/9 mm. $[\alpha]_D^{20}$ − 15·54° in H_2O. *Triacetyl*: m.p. 86–7°. $[\alpha]_D^{16}$ − 53·5° in $C_2H_2Cl_4$.

3 : 4-*Di-Me ether*: cryst. from Et_2O. M.p. 102–3°. n_D^{19} 1·4730. $[\alpha]_D^{19}$ + 20° in H_2O.

2 : 3 : 4-*Tri-Me ether*: syrup. B.p. 141°/19 mm. n_D^{15} 1·4565. $[\alpha]_D^{18}$ + 24·15° ⟶ + 25·44° in H_2O, + 3·25° ⟶ + 5·82° in C_6H_6, − 4·86° ⟶ − 9·52° in EtOH. *Phenylhydrazone*: yellow prisms. M.p. 126–8° decomp. *Anilide*: cryst. from pet. ether. M.p. 112°.

Tetranitrate: cryst. from EtOH. M.p. 135° decomp.

β-*Form*:

Needles from Me_2CO. M.p. 123·5–124·5°. $[\alpha]_D$ + 38·4° ⟶ + 8·9° in H_2O. Sol. H_2O, EtOH.

Methylglycoside: needles from AcOEt. M.p. 138–40°. $[\alpha]_D^{20}$ + 95·39° in H_2O. *Triacetyl*: m.p. 151–2°. $[\alpha]_D^{18}$ + 45·73° in $C_2H_2Cl_4$.

Triacetyl: plates from Et_2O. M.p. 96–8°. $[\alpha]_D^{21}$ + 28·09° ⟶ + 18·6° in EtOH.

Tetra-acetyl: m.p. 98·5–99°. $[\alpha]_D^{20}$ + 13·3° in $CHCl_3$.

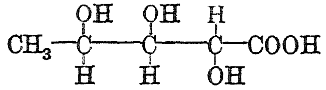
dl-.

M.p. anhyd. 151·3–153°.

Votoček, Valentin, Rac, *Chem. Abstracts*, 1931, **25**, 84.
Votoček, Valentin, *Compt. rend.*, 1926, **183**, 62.
Fischer, Zach, *Ber.*, 1912, **45**, 3770.
Harding, *Sugar*, 1923, **25**, 82.
Hirst, Macbeth, *J. Chem. Soc.*, 1926, 22.

Clark, *J. Biol. Chem.*, 1919, **38**, 255.
Walton, *J. Am. Chem. Soc.*, 1921, **43**, 127.
Fischer, Bergmann, Rabe, *Ber.*, 1920, **53**, 2362.
Jackson, Hudson, *J. Am. Chem. Soc.*, 1937, **59**, 1076.

Rhamnotetronic Acid (*Methyltetronic acid*)

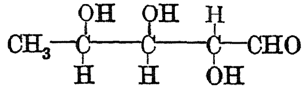


$C_5H_{10}O_5$ MW, 150

l-.
Free acid not isolated.
Brucine salt: needles from EtOH. M.p. 145–50° decomp.
Amide: $C_5H_{11}O_4N$. MW, 149. Plates from EtOH. M.p. 135° decomp. $[\alpha]_D$ + 54·8° in H_2O.
Phenylhydrazide: laminæ from AcOEt. M.p. 169°.
γ-Lactone: $C_5H_8O_4$. MW, 132. Needles from H_2O or EtOH. M.p. 123° (120–1°). $[\alpha]_D^{20}$ – 47·5° in H_2O. Sol. EtOH, AcOEt, Et_2O. Spar. sol. $CHCl_3$, C_6H_6.

Ruff, *Ber.*, 1902, **35**, 2365.
Hudson, Chernoff, *J. Am. Chem. Soc.*, 1918, **40**, 1005.

Rhamnotetrose (*Methyltetrose*)



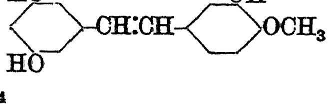
$C_5H_{10}O_4$ MW, 134

l-.
Syrup. Sol. H_2O, EtOH. $[\alpha]_D^{20}$ – 30·5° ⟶ – 16·35° in 96% EtOH. Reduces Fehling's. HNO_3 ⟶ tartaric acid.
Benzylphenylhydrazone: needles from C_6H_6. M.p. 96–7°. $[\alpha]^{20}$ – 6·5° in 96% EtOH. Sol. EtOH, Et_2O. Prac. insol. H_2O, C_6H_6.
Phenylosazone: yellow needles from EtOH. M.p. 172–3°. Sol. hot EtOH, C_6H_6. Prac. insol. H_2O, Et_2O.

Ruff, *Ber.*, 1902, **35**, 2364.
Fischer, *Ber.*, 1896, **29**, 1381.

Rhamnoxanthin.
See Frangulin.

Rhapontigenin (3 : 4 : 3′ : 5′-*Tetrahydroxy stilbene* 4-*methyl ether*)



$C_{15}H_{14}O_4$ MW, 258

Aglucone from rhapontin from rhapontic spice. M.p. 186–7°.
Triacetyl: (1) M.p. 114°. (2) M.p. 128°.
Tribenzoyl: m.p. 142°.

Kawamura, *J. Pharm. Soc. Japan*, 1938, **58**, 405.
Takaoka, *Proc. Imper. Acad., Tokyo*, 1940, **16**, 408.

Rhapontin

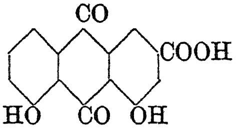
$C_{21}H_{24}O_9$ MW, 420

Glycoside from rhapontic spice. Decomp. at 236–7°. $[\alpha]_D^{32}$ – 59·5° in Me_2CO. Hyd. ⟶ glucose + rhapontigenin.
Acetyl deriv.: m.p. 135–6°.

Kawamura, *J. Pharm. Soc. Japan*, 1938, **58**, 405.

Rheic Acid.
See Chrysophanic Acid.

Rhein (*Chrysazin*-3-*carboxylic acid*, 4 : 5-*dihydroxyanthraquinone*-2-*carboxylic acid*)



$C_{15}H_8O_6$ MW, 284

Occurs in senna leaves. Yellow needles from MeOH. M.p. 321°. Sol. Py. Spar. sol. EtOH, Et_2O, C_6H_6, AcOH, Me_2CO, $CHCl_3$, pet. ether.
Diacetyl: m.p. 258° (246° decomp.).
Mono-propionyl deriv.: m.p. 223–4°. *Me ether*: m.p. 281–2°.
Dipropionyl: m.p. 223–4°.
Dibenzoyl: m.p. 262° (253–5°).
Me ester: $C_{16}H_{10}O_6$. MW, 298. Orange needles from MeOH. M.p. 174°.
Et ester: $C_{17}H_{12}O_6$. MW, 312. Orange needles from EtOH. M.p. 159° (160–1°). *Diacetyl*: m.p. 170°.
Propyl ester: $C_{18}H_{14}O_6$. MW, 326. Brown needles. M.p. 145°. *Diacetyl*: m.p. 178°.
Isopropyl ester: brown cryst. M.p. 181°. *Diacetyl*: m.p. 190°.
Isobutyl ester: $C_{19}H_{16}O_6$. MW, 340. Yellow needles. M.p. 153°. *Diacetyl*: m.p. 109°.
Phenyl ester: $C_{21}H_{12}O_6$. MW, 360. Yellow needles. M.p. 215°. *Diacetyl*: m.p. 170°.
Benzyl ester: $C_{22}H_{23}O_6$. MW, 374. *Diacetyl*: m.p. 203°.
Di-Me ether: $C_{17}H_{12}O_6$. MW, 312. Brown needles from EtOH. M.p. 283–4°. *Et ester*: $C_{19}H_{16}O_6$. MW, 340. Yellow needles from AcOH. M.p. 185–7°. *Chloride*: $C_{17}H_{11}O_5Cl$. MW, 330·5. Yellow prisms from $CHCl_3$–pet. ether. M.p. 190°. *Amide*: $C_{17}H_{13}O_5N$. MW, 311. Brownish-yellow plates from AcOEt. M.p. 287°.

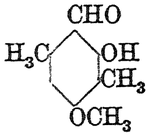
Oesterle, Haugseth, *Arch. Pharm.*, 1915, **253**, 330.

Rheochrysidine.
See Physcione.

Rheumatin.
See under Quinine.

Rheum-emodin.
See Frangula-emodin.

Rhizonaldehyde (*2-Hydroxy-4-methoxy-3 : 6-dimethylbenzaldehyde, β-orcylaldehyde 5-methyl ether*)



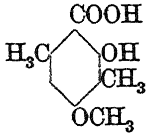
$C_{10}H_{12}O_3$ MW, 180

Prisms from EtOH.Aq. M.p. 136°. Sol. Et_2O, C_6H_6, hot EtOH, hot pet. ether. $FeCl_3$ ⟶ reddish-brown col.

Oxime: m.p. 188–9°.
Acetyl: m.p. 71°.
Carbomethoxyl: m.p. 90°.

Sonn, *Ber.*, 1916, **49**, 2591.
Robertson, Stephenson, *J. Chem. Soc.*, 1930, 318.
Pfau, *Helv. Chim. Acta*, 1928, **11**, 873.

Rhizonic Acid (*2-Hydroxy-4-methoxy-3 : 6-dimethylbenzoic acid*)



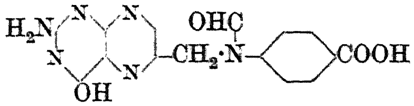
$C_{10}H_{12}O_4$ MW, 196

Occurs in *Evernii prunastri*, and *Rizocarpon geographicum*, Linn. Cryst. from EtOH. M.p. 235° (232°).

Me ester: $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH. M.p. 95°. *Me ether*: $C_{12}H_{16}O_4$. MW, 224. B.p. 161–3°/10 mm.
Et ester: $C_{12}H_{16}O_4$. MW, 224. Needles from EtOH. M.p. 82° (81°).
Acetyl: m.p. 146° decomp.
2-Me ether: $C_{11}H_{14}O_4$. MW, 210. Prisms from pet. ether. M.p. 104–5°.

Asahina, Akagi, *Ber.*, 1935, **68**, 1132.
Robertson, Stephenson, *J. Chem. Soc.*, 1930, 319.
Sonn, *Ber.*, 1929, **62**, 3012.

Rhizopterin (*12-Formylpteroic acid*)



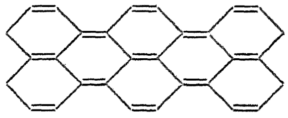
$C_{15}H_{12}N_6O_4$ MW, 340

Occurs in *Rhizopus nigricans*. Pale yellow leaflets from AcOH.Aq. Darkens at 285°. Does not melt below 300°. Insol. H_2O and usual org. solvents. Sol. acids and alkalis. 1% HCl ⟶ *p*-aminobenzoic acid. Biologically active.

Acetyl: white cryst. from AcOH.Aq. Does not melt below 300°.
Benzoyl: white cryst. from AcOH.Aq. M.p. 250° decomp.
Me ether: needles from AcOH.Aq. M.p. 258–68° with effervescence.

Mozingo, Folkers *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2753.

Rhodacene (*Quinonoid form of chalkacene,* q.v.)



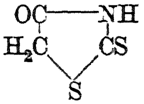
$C_{30}H_{16}$ MW, 376

Violet microcryst. M.p. 338–40°. Sol. C_6H_6 and homologues with red col. and red fluor.

Dziewoński, Podgorska, Lemberger, Suszka, *Ber.*, 1920, **53**, 2173.

Rhodanic Acid.
See Rhodanine.

Rhodanine (*Rhodanic acid*)



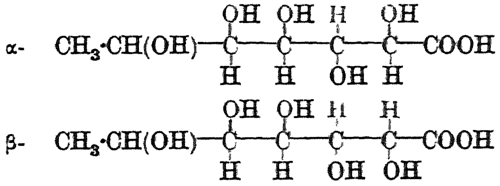
$C_3H_3ONS_2$ MW, 133

Pale yellow prisms from EtOH. M.p. 170°.

Julian, Sturgis, *J. Am. Chem. Soc.*, 1935, **57**, 1126.

Rhodeitol.
See Fucitol.

Rhodeohexonic Acid



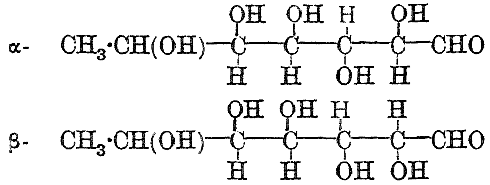
$C_7H_{14}O_7$ MW, 210

α-Form:
Syrup rapidly changing to lactone.
γ-Lactone: $C_7H_{12}O_6$. MW, 192. Cryst. + $2H_2O$. M.p. 41·5° (129–31°).
Amide: $C_7H_{15}O_6N$. MW, 209. M.p. 206°.
Phenylhydrazide: m.p. 231° decomp.

β-Form:
Syrup rapidly changing to lactone.
γ-Lactone: m.p. 115°.
Amide: amorph. grey powder. M.p. 197–8°.
Phenylhydrazide: m.p. 211° decomp.

Votoček, *Chem. Abstracts*, 1935, **29**, 2918; *Ber.*, 1910, **43**, 469.
Krauz, *Ber.*, 1910, **43**, 482.

Rhodeohexose



$C_7H_{14}O_6$ MW, 194

α-Form:
Cryst. from EtOH. M.p. 125–6°. $[\alpha]_D^{20}$ + 11·96° in H_2O.
Phenylhydrazone: m.p. 150°.
p-*Bromophenylhydrazone*: m.p. 173°.

β-Form:
Syrup.
Phenylhydrazone: m.p. 131–7°.
p-*Bromophenylhydrazone*: m.p. 145°.

Krauz, *Chem. Zentr.*, 1911, II, 1216; *Ber.*, 1910, **43**, 482.

Rhodeoretin.
See Convolvulin.

Rhodeose.
See Fucose.

Rhodinal.
See Citronellal.

Rhodinic Acid.
See Citronellic Acid.

Rhodinol.
See Citronellol.

Rhodinolic Acid.
See Citronellic Acid.

Rhodopsin.
See Visual Purple.

Rhodoviolascin

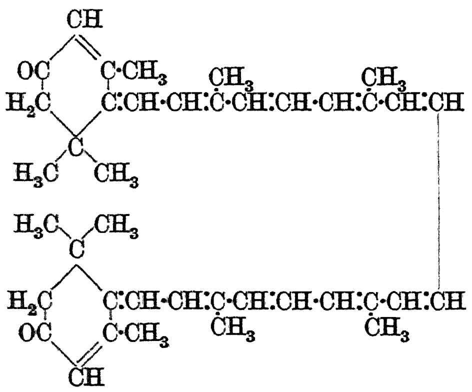
$C_{40}H_{54}(OCH_3)_2$

$C_{42}H_{60}O_2$ MW, 596

Carotinoid pigment from Rhodovibrio bacteria. Cryst. from C_6H_6. M.p. 218°. Contains 13 double bonds.

Karrer, Solmssen, *Helv. Chim. Acta*, 1936, **19**, 3.

Rhodoxanthin



$C_{40}H_{50}O_2$ MW, 562

Pigment occurring in *Taxus baccata*, Linn. (Yew). Bluish-black leaflets from C_6H_6–MeOH. M.p. 219°. Absorption maxima in CS_2 at 564, 525 and 491 mμ.
Dioxime: m.p. 227–8°. Absorption maxima in CS_2 at 516, 483 and 453 mμ.

Kuhn, Brockmann, *Ber.*, 1933, **66**, 828.

Rhœadine

$C_{21}H_{21}O_6N$ MW, 383

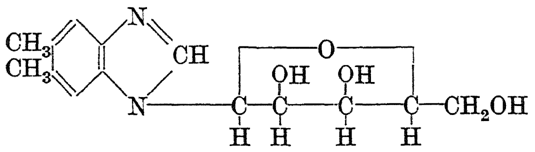
Occurs in *Papaver rhœas* Linn. (red poppy). Colourless needles from $CHCl_3$–Et_2O or EtOH. M.p. 256–7°. $[\alpha]_D^{19}$ + 243° in $CHCl_3$. Spar. sol. Et_2O, pet. ether, $CHCl_3$, EtOH, H_2O. Slightly sol. AcOEt, methylene dichloride. Salts difficult to prepare. Decomp. by min. acids. Contains one methoxy group, two methylenedioxy groups and one CH_3N group.

Späth, *Monatsh.*, 1936, **68**, 33.
Awe, *Arch. Pharm.*, 1941, **279**, 116.

Rhombinine.
See Anagyrine.

Ribal.
See Arabinal.

Ribazole (1-D-*Ribofuranosido*-5 : 6-*dimethylbenziminazole*)



$C_{14}H_{18}O_4N_2$ MW, 278

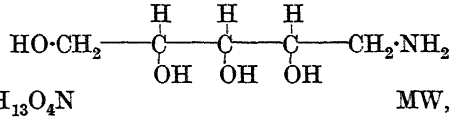
α-.
Degradation product of vitamin B_{12}.
Picrate: cryst. M.p. 213–14°. $[\alpha]_D^{23}$ + 9·9° ± 1·6° in Py.
2′(or 3′)-*Phosphate*: m.p. 240–1° decomp.
Dibrucine salt: m.p. 169–75°.

β-.
Cryst. from MeOH. M.p. 202–3°. $[\alpha]_D$ —45·3° ± 2° in Py.
Picrate: m.p. 192° (175–7°). $[\alpha]_D^{23}$ —24° ± 2° in Py.

Folkers *et al.*, *J. Am. Chem. Soc.*, 1950, **72**, 1866; 1952, **74**, 5549.
Weygand, Wirth, *Ber.*, 1952, **85**, 1001.

Ribitol.
See Adonitol.

Ribitylamine (*Ribamine*)



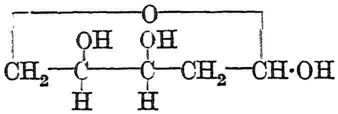
$C_5H_{13}O_4N$ MW, 151

d-.
M.p. 146°. $[\alpha]_D^{22}$ — 31° in Py.
B,HCl: needles from HCl–Me_2CO. M.p. 129°. $[\alpha]_D^{20}$ — 12° in H_2O. Sol. H_2O. Spar. sol. Me_2CO, EtOH.

Penta-acetyl: needles from Me_2CO. M.p. 114–15°.
N-Phenyl: m.p. 125–7°. $[\alpha]_D^{25}$ − 43° in Py.
N-4 : 5-*Dimethyl-2-*(p-*nitrophenylazo)-phenyl*: brick-red cryst. from MeOH. M.p. 180–2°. *Tetra-acetyl*: cryst. from EtOH. M.p. 168–9°.
l-.
N-3 : 4-*Dimethylphenyl*: cryst. from MeOH. M.p. 142–3°. $[\alpha]_D^{20}$ + 3·4°.

Kuhn, Wendt, *Ber.*, 1948, **81**, 553.
Karrer *et al.*, *Helv. Chim. Acta*, 1935, **18**, 908.
Karrer, Meerwein, *Helv. Chim. Acta*, 1936, **19**, 1190.
Friedrich, Weygand, *Ber.*, 1940, **73**, 1259.
Tishler *et al.*, *J. Am. Chem. Soc.*, 1945, **67**, 2165.
Bergel, Cohen, Haworth, *J. Chem. Soc.*, 1945, 165.

2-Ribodesose (2-*Deoxyribose*)

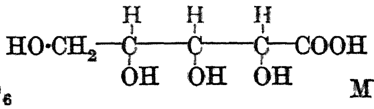


$C_5H_{10}O_4$ MW, 134

d-. (Thyminose.)
Constituent of many nucleic acids. Cryst. from isopropyl alcohol. M.p. 87–90°. $[\alpha]_D^{22}$ −55·2° (equilibrium) in H_2O.
Anil: cryst. M.p. 174–5°. $[\alpha]_D^{22}$ +19·5° (equilibrium) in H_2O.
Benzylphenylhydrazone: cryst. M.p. 128°. $[\alpha]_D^{25}$ −17·5° in Py.
l-. (*l*-Arabodesose.)
Cryst. Sinters at 67°, m.p. 80°. $[\alpha]_D^{25}$ +91·7° (initial) ⟶ + 40·5° (final) in Py.
Benzylphenylhydrazone: m.p. 125–6°. $[\alpha]_D^{25}$ + 17·5° in Py.

Levene, Mikeska, Mori, *J. Biol. Chem.*, 1930, **85**, 785.
Overend, Stacey, Wiggins, *J. Chem. Soc.*, 1949, 1363.
Hough, *Chemistry and Industry*, 1951, 406.

3-Ribodesose.
See 3-Xylodesose.
Riboflavine.
See Lactoflavine.
Ribonic Acid

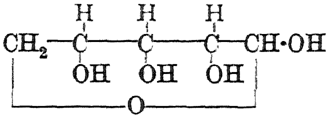


$C_5H_{10}O_6$ MW, 166

d-.
Cryst. from MeOH. M.p. 112–13°. $[\alpha]_D^{25}$ −17·0° in H_2O, − 17·3° in MeOH.
γ-Lactone: $C_5H_8O_5$. MW, 148. Cryst. from AcOEt. M.p. 72–8° (80°). $[\alpha]_D$ + 18·4° in H_2O. *Tri-acetyl*: cryst. from AcOH.Aq. M.p. 54–6°. $[\alpha]_D^{25}$ + 27° in $CHCl_3$.
Hydrazide: m.p. 150°. $[\alpha]_D^{15}$ + 27·5°.
Tetra-acetyl nitrile: cryst. from Et_2O or MeOH. M.p. 71–2°. $[\alpha]_D^{25}$ + 34·5° in $CHCl_3$.
l-.
M.p. 104–5°. $[\alpha]_D^{20}$ + 17·6° in H_2O.
γ-Lactone: prisms from AcOEt. M.p. 84–6° (80°, 72–6°). Sol. H_2O, EtOH, Me_2CO. Mod. sol. AcOEt. Spar. sol. Et_2O. $[\alpha]_D^{20}$ − 18° in H_2O.
Amide: $C_5H_{11}O_5N$. MW, 165. Plates. M.p. 136–7°. $[\alpha]_D^{14}$ − 18·7°.
Phenylhydrazide: m.p. 204°. $[\alpha]_D^{15}$ − 33° in H_2O.

Schmidt, Weber-Molster, *Ann.*, 1934, **515**, 43.
Rehorst, *Ann.*, 1933, **503**, 143.
Fischer, Piloty, *Ber.*, 1891, **24**, 4216.
Hasenfratz, *Compt. rend.*, 1927, **184**, 210.
v. Ekenstein, Blanksma, *Chem. Zentr.*, 1913, II, 1562.
Ladenburg, Tishler, Wellman, Babson, *J. Am. Chem. Soc.*, 1944, **66**, 1217.

Ribose



$C_5H_{10}O_5$ MW, 150

d-.
A constituent of many nucleosides and nucleotides. Cryst. M.p. 95° (86–7°). Hygroscopic. $[\alpha]_D$ − 21·5° (− 19·5°) in H_2O. $[\alpha]_D^{20}$ − 38·2° ⟶ 43·1° (2 days) in Py. Sol. H_2O. Spar. sol. EtOH.
p-*Bromophenylhydrazone*: cryst. M.p. 170° (166–7°). $[\alpha]_D^{25}$ + 10·3° in EtOH, − 19·3° in Py.
Phenylosazone: yellow cryst. from H_2O. M.p. 160° (162–3°, 163–4°). Indentical with phenylosazone of *d*-arabinose.
p-*Bromophenylosazone*: m.p. 180–5°.
Benziminazole deriv.: m.p. 190°. $[\alpha]_D^{25}$ + 22·5° in 0·1N-HCl. *B,HCl*: m.p. 196–8°. *Picrate*: m.p. 185–6°.
β-*Methylpyranoside*: m.p. 83°. $[\alpha]_D^{20}$ − 105° in H_2O.
Methylfuranoside: b.p. 150°/0·01 mm. n_D^{15} 1·488. $[\alpha]_D^{15}$ + 13°. *Tri-Me ether*: b.p. 133°/15 mm. n_D^{15} 1·435. $[\alpha]_D^{18}$ + 59°.
3 : 5-*Di-Me ether phenylosazone*: yellow needles from EtOH. M.p. 161°.
2 : 3 : 4-*Tri-Me ether*: m.p. 98–100°.
2 : 3 : 5-*Tri-Me ether*: b.p. 97°/0·01 mm. $[\alpha]_D^{24}$ + 41·4°. n_D^{20} 1·4523. *Anilide*: cryst. from Et_2O–pet. ether. M.p. 56·5°.
2 : 3 : 4-*Triacetyl*: syrup. $[\alpha]_D^{20}$ − 26·3° in H_2O.
2 : 3 : 5-*Triacetyl*: hygroscopic syrup. $[\alpha]_D^{20}$ − 24·2° in H_2O.

1 : 2 : 3 : 5-*Tetra-acetyl*: b.p. 100–110°/10^{-4} mm. $[\alpha]_D^{20}$ − 3·6° in MeOH, $[\alpha]_D^{15}$ +20° in $CHCl_3$.

l-.

Cryst. from EtOH. M.p. 87°. $[\alpha]_D$ + 18·8° in H_2O. Sol. H_2O. Spar. sol. EtOH. Sweet taste.

Phenylhydrazone: cryst. from EtOH. M.p. 154–5° decomp. Sol. H_2O. Spar. sol. EtOH.

p-*Bromophenylhydrazone*: cryst. from EtOH. M.p. 164–5°. Sol. H_2O.

Phenylosazone: yellow cryst. from Me_2CO or H_2O. M.p. 166°. Identical with phenylosazone of *l*-arabinose. $[\alpha]_D$ + 1° in Py–EtOH, + 18·9° in EtOH. Sol. hot H_2O, EtOH, Me_2CO, Py. Insol. H_2O, Et_2O, C_6H_6, ligroin.

dl-.

M.p. 83–4°.

Phenylosazone: yellow needles or prisms. M.p. 166–8°. Identical with phenylosazone of *dl*-arabinose.

Austin, Humoller, *J. Am. Chem. Soc.*, 1934, **56**, 1152.
Levene, *J. Biol. Chem.*, 1935, **108**, 419.
Steiger, *Helv. Chim. Acta*, 1936, **19**, 189.
Fischer, Piloty, *Ber.*, 1891, **24**, 4220.
Levene, Jacobs, *Ber.*, 1909, **42**, 3247.
v. Ekenstein, Blanksma, *Chem. Zentr.*, 1913, II, 1562; 1914, I, 965.
Minsaas, *Ann.*, 1934, **512**, 286.
Jackson, Hudson, *J. Am. Chem. Soc.*, 1941, **63**, 1229.
Dimler, Link, *J. Biol. Chem.*, 1943, **150**, 345.
Berger *et al.*, *J. Org. Chem.*, 1946, **11**, 91.
Howard, Lythgoe, Todd, *J. Chem. Soc.*, 1947, 1052.
Barker, *J. Chem. Soc.*, 1948, 2035.
Bredereck, Hoepfner, *Ber.*, 1948, **81**, 51.

Ribulose.

See Adonose.

Ricinelaidic Acid (*Stereoisomer of ricinoleic acid*)

$CH_3\cdot[CH_2]_5\cdot CH(OH)\cdot CH_2\cdot CH{:}CH\cdot[CH_2]_7\cdot COOH$

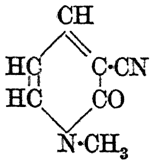
$C_{18}H_{34}O_3$ MW, 298

Needles from ligroin. M.p. 51–2°. B.p. 240–2°/10 mm. Sol. EtOH. $[\alpha]_D^{20}$ + 6·67° in EtOH.

Et ester: $C_{20}H_{38}O_3$. MW, 326. M.p. 16°.
Amide: $C_{18}H_{35}O_2N$. MW, 297. M.p. 91–3°.
Phenylhydrazide: m.p. 110–110·5°.

Mühle, *Ber.*, 1913, **46**, 2096.
Krafft, *Ber.*, 1888, **21**, 2735.

Ricinidine

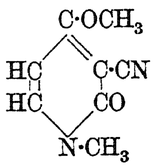


$C_7H_6ON_2$ MW, 134

M.p. 140°. B.p. 243°/28 mm.

Späth, Koller, *Ber.*, 1923, **56**, 886.

Ricinine (4-*Methoxy*-2-*keto*-1-*methyl*-1 : 2-*dihydronicotinonitrile*, 4-*methoxy*-N-*methyl*-3-*cyano*-2-*pyridone*)



$C_8H_8O_2N_2$ MW, 164

Occurs in castor-oil seeds. Leaflets or prisms from H_2O. M.p. 201·5°. Sol. hot H_2O, hot $CHCl_3$. Spar. sol. EtOH. Insol. pet. ether. Reduces $KMnO_4$.

B,HgCl₂: m.p. 204°.

Späth, Koller, *Ber.*, 1925, **58**, 2124.
Schroeter, Seidler, Sulzbacher, Kametz, *Ber.*, 1932, **65**, 432.
Reitmann, *Chem. Abstracts*, 1935, **29**, 4359.

Ricinoleic Acid (*Ricinolic acid*)

$CH_3\cdot[CH_2]_5\cdot CH(OH)\cdot CH_2\cdot CH{:}CH\cdot[CH_2]_7\cdot COOH$

$C_{18}H_{34}O_3$ MW, 298

Occurs in castor oil as glyceride. B.p. 226–8°/10 mm. (245°/10 mm.). Sol. EtOH, Et_2O. $[\alpha]_D^{22}$ + 5·05°. D^{15} 0·9496. n_D^{15} 1·4145.

Me ester: $C_{19}H_{36}O_3$. MW, 312. B.p. 225–7°/15 mm. *Acetyl*: b.p. 210°/13 mm. D^{22} 0·9301. n_D^{22} 1·4570. $[\alpha]_D^{22}$ + 15·25°. *Propionyl*: b.p. 260°/13 mm. D^{18} 0·9226. n_D^{18} 1·4535. $[\alpha]_D^{18}$ + 16·88°. *Benzoyl*: b.p. 195–6°/0·08 mm.

Et ester: $C_{20}H_{38}O_3$. MW, 326. B.p. 258°/13 mm. D^{22} 0·9145. n_D^{22} 1·4618. $[\alpha]_D^{22}$ + 5·28°. *Acetyl*: b.p. 255–6°/13 mm. D^{22} 0·9170. n_D^{22} 1·4540. $[\alpha]_D^{22}$ + 14·85°. *Propionyl*: b.p. 265°/13 mm. D^{18} 0·9151. n_D^{18} 1·4517. $[\alpha]_D^{18}$ + 16·06°.

Propyl ester: $C_{21}H_{40}O_3$. MW, 340. B.p. 268°/13 mm. D^{22} 0·9079. n_D^{22} 1·4573. $[\alpha]_D^{22}$ + 4·15°. *Acetyl*: b.p. 260°/13 mm. D^{22} 0·9117. n_D^{22} 1·4513. $[\alpha]_D^{22}$ + 14·40°. *Propionyl*: b.p. 310–20°/645 mm. D^{22} 0·9128. n_D^{22} 1·4498. $[\alpha]_D^{22}$ + 13·61°.

Isopropyl ester: b.p. 210°/10 mm. D^{22} 0·9083. n_D^{22} 1·4583. $[\alpha]_D^{22}$ + 4·04°.

Butyl ester: $C_{22}H_{42}O_3$. MW, 354. B.p. 278°/13 mm. D^{22} 0·9058. n_D^{22} 1·4566. $[\alpha]_D^{22}$ + 3·73°. *Acetyl*: b.p. 249–58°/25 mm., 240–5°/18 mm.

Isobutyl ester: b.p. 282°/9 mm. D^{22} 0·9028. n_D^{22} 1·4538. $[\alpha]_D^{22}$ + 4·01°. *Acetyl*: b.p. 255–60°/13 mm. D^{22} 0·9012. n_D^{22} 1·4548. $[\alpha]_D^{22}$ + 9·58°. *Propionyl*: b.p. 325–35°/160 mm. D^{22} 0·9027. n_D^{22} 1·4525. $[\alpha]_D^{22}$ + 9·45°.

Heptyl ester: $C_{25}H_{48}O_3$. MW, 396. B.p. 295°/10 mm. D^{22} 0·8983. n_D^{22} 1·4566. $[\alpha]_D^{22}$ + 3·32°.

Phenylhydrazide: m.p. 63°.

van Alphen, *Rec. trav. chim.*, 1925, **44**, 1064.
Ricler, U.S.P., 1,955,021, (*Chem. Abstracts*, 1934, **28**, 2926).
Straus, Heinze, Salzmann, *Ber.*, 1933, **66**, 631.
Böhme, D.R.P., 592,053, (*Chem. Abstracts*, 1934, **28**, 2725).
Rutovskii, Ostroumova, *Chem. Abstracts*, 1940, **34**, 2332.

Ricinolic Acid.
See Ricinoleic Acid.

Ricinstearolic Acid

$CH_3{\cdot}[CH_2]_5{\cdot}CH(OH){\cdot}CH_2{\cdot}C{:}C{\cdot}[CH_2]_7{\cdot}COOH$

$C_{18}H_{32}O_3$ MW, 296

Needles from ligroin. M.p. 52°. B.p. 260°/10 mm. Sol. EtOH, Et_2O. Insol. H_2O. $[\alpha]_D$ + 13·67° in H_2O.

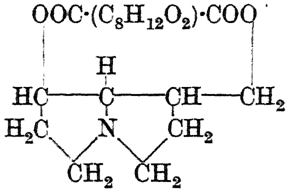
A_2,Ba: m.p. 135°.

Me ester: $C_{19}H_{34}O_3$. MW, 310. B.p. 225°/12 mm.

Et ester: $C_{20}H_{36}O_3$. MW, 324. B.p. 230°/12 mm.

Mühle, *Ber.*, 1913, **46**, 2091.

Riddelliine



$C_{18}H_{23}O_6N$ MW, 349

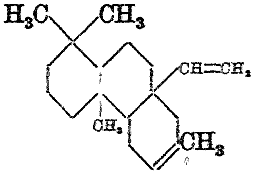
Occurs in *Senecio Riddellii*. Cryst. from EtOH. M.p. 197–8°. $[\alpha]_D^{25}$ − 109·5° in $CHCl_3$. $Ba(OH)_2$ ⟶ riddellic acid, m.p. 62°, $[\alpha]_D^{31}$ − 1·96°.

B,HCl: cryst. from EtOH. M.p. 225–6° decomp. $[\alpha]_D^{25}$ − 80·6° in H_2O.

Methiodide: cryst. from H_2O. M.p. 260–2° decomp.

Adams *et al.*, *J. Am. Chem. Soc.*, 1942, **64**, 2760.

Rimuene



Suggested formula

$C_{20}H_{32}$ MW, 272

From leaves of *Dacrydium cupressinum* (Salander), etc. Cryst. from EtOH. M.p. 55°. $[\alpha]_D$ + 44·7° in $CHCl_3$. Se dehydrogenation ⟶ pimanthrene.

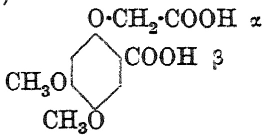
Hydrochloride: $C_{20}H_{33}Cl$. M.p. 63°.

Nitroso-chloride: $C_{20}H_{32}ONCl$. M.p. 86–8°.

Tetrabromide: m.p. 55–60°.

McDowall, Finlay, *J. Soc. Chem. Ind.*, 1925, **44**, 42T.
Beath, *J. Soc. Chem. Ind.*, 1933, **52**, 338T.
Brandt, *Chem. Abstracts*, 1939, **33**, 551.

Rissic Acid (4 : 5-*Dimethoxy*-2-*carboxyphenoxyacetic acid*)



$C_{11}H_{12}O_7$ MW, 256

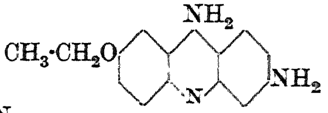
Needles from hot butyl alcohol. M.p. 262° (256° decomp.).

α : β-*Di-Me ester*: $C_{13}H_{16}O_7$. MW, 284. M.p. 86°. B.p. 265–70°/0·4 mm.

α-*Et ester*: $C_{13}H_{16}O_7$. MW, 284. Prisms from EtOH. M.p. 190°.

Robertson, *J. Chem. Soc.*, 1932, 1380.
Butenandt, McCartney, *Ann.*, 1932, **494**, 17.
Clark, *J. Am. Chem. Soc.*, 1931, **53**, 2371; 1932, **54**, 2548.
Takei, Miyajima, Ono, *Ber.*, 1931, **64**, 251; *Chem. Abstracts*, 1932, **26**, 5300.

Rivanol (7-*Ethoxy*-2 : 5-*diaminoacridine*)



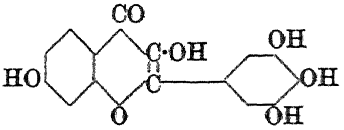
$C_{15}H_{15}ON_3$ MW, 253

Orange-yellow cryst. from 50% EtOH. M.p. 226°. Antiseptic.

Albert, Gledhill, *J. Soc. Chem. Ind.*, 1942, **61**, 159.

Robigenin.
See Kæmpferol.

Robinetin (3 : 7 : 3′ : 4′ : 5′-*Pentahydroxyflavone*)



$C_{15}H_{10}O_7$ MW, 302

Found in stem-wood of *Robinia pseudacacia*, Linn., and *Gleditschia monosperma*, Walt. Greenish-yellow needles from AcOH.Aq. Decomp. at 325–30°. Sol. EtOH, Me_2CO, AcOH, AcOEt, Py. Spar. sol. H_2O, Et_2O. Insol. $CHCl_3$, C_6H_6, pet. ether. Conc. H_2SO_4 ⟶ yellow sol. Alkalis ⟶ red sols. Drop of conc. H_2SO_4 in red AcOH sol. gives characteristic intense green col.

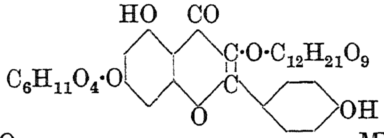
3 : 3′ : 4′ : 5′-*Tetra-Me ether*: $C_{19}H_{18}O_7$. MW, 358. Pale yellow cryst. from EtOH. M.p. 250–1°. Alkalis and conc. H_2SO_4 ⟶ yellow sols. *Acetyl*: needles from EtOH. M.p. 149–50°.

Penta-Me ether: $C_{20}H_{20}O_7$. MW, 372. Prisms from MeOH. M.p. 149°. Conc. HCl ⟶ yellow sol.

Penta-acetyl: cryst. from EtOH. M.p. 224°. Sol. EtOH, Me_2CO, C_6H_6.

Charlesworth, Robinson, *J. Chem. Soc.*, 1933, 269.

Robinin



$C_{33}H_{40}O_{19}$ MW, 740

Found in flowers of *Robinia pseudoacacia*, Linn. Exists in two forms. α: cryst. + $8H_2O$ from hot H_2O. M.p. (after drying) 195–7°. β: cryst. from EtOH. M.p. 249—50°. Hyd. to kaempferol, galactose (1 mol.) and rhamnose (2 mol.)

Sando, *J. Biol. Chem.*, 1932, **94**, 675.
Zemplén, Bognár, *Ber.*, 1941, **74**, 1784.

Roccellic Acid (*1-Methyl-2-dodecylsuccinic acid*)

CH_3·CH·COOH
CH_3·$[CH_2]_{11}$·CH·COOH

$C_{17}H_{32}O_4$ MW, 300

Occurs in *Lecanora sordida*. White plates from pet. ether. M.p. 131—2°. $[\alpha]_D$ + 17·4° in EtOH. Heat to 160° ⟶ anhydride.

Di-Me ester: cryst. from Et_2O. M.p. 28–9°.

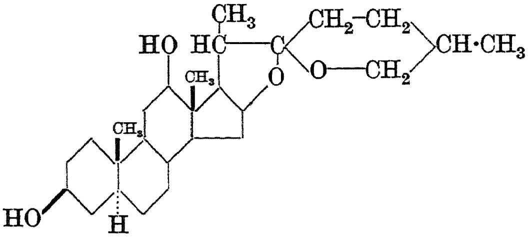
Phenylimide: cryst. from MeOH. M.p. 57–8°.

Kennedy, Breen, Keane, Nolan, *Chem. Zentr.*, 1938, I, 3062.

Rochelle Salt.

See under Tartaric Acid.

Rockogenin



$C_{27}H_{44}O_4$ MW, 432

Thick needles from MeOH, m.p. 208–10°; cryst. from Et_2O, m.p. 217–20°.

3-*Acetyl*: m.p. 211–13°.

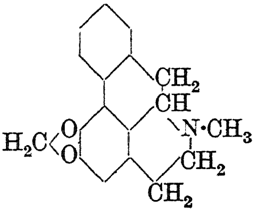
3 : 12-*Diacetyl*: m.p. 203–5°.

Marker *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2167.
Hirschmann, Snoddy, Wendler, *J. Am. Chem. Soc.*, 1952, **74**, 2693.

Rodinal.

See p-Aminophenol.

Roemerine (*Remerine*)



$C_{18}H_{17}O_2N$ MW, 279

Alkaloid from *Roemaria refracta*, D.C. M.p. 102–3°. $[\alpha]_D$ − 77·18° in EtOH.

B,HCl: m.p. 262–3°.

Methiodide: m.p. 215–16°.

Picrate: m.p. 195–6°.

Konovalova, Junusov, Orékhov, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 1868; *Bull. soc. chim.*, 1939, **6**, 1479; 1940, **7**, 70.
Marion, Grassie, *J. Am. Chem. Soc.*, 1944, **66**, 1290.

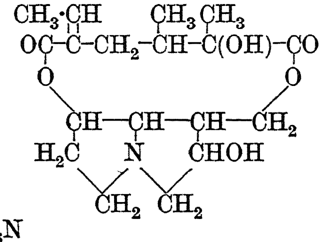
Rongalite.

See under Formaldehyde.

Rosilic Acid.

See 9-Hydroxystearic Acid.

Rosmarinine



$C_{18}H_{27}O_6N$ MW, 353

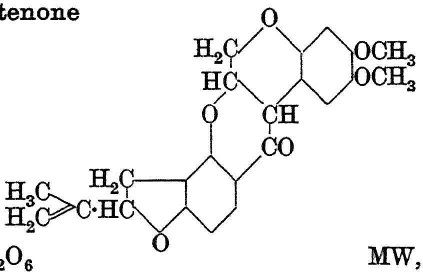
Alkaloid from *Senecio rosmarinifolius*, Linn. Shining laminæ from Me_2CO or EtOH. M.p. 209°. $[\alpha]_D^{24}$ − 91·5° in MeOH.

Richardson, Warren, *J. Chem. Soc.*, 1943, 452.
Kropman, Warren, *J. Chem. Soc.*, 1950, 700.

Rotenic Acid.

See Isotubaic Acid.

Rotenone



$C_{23}H_{22}O_6$ MW, 394

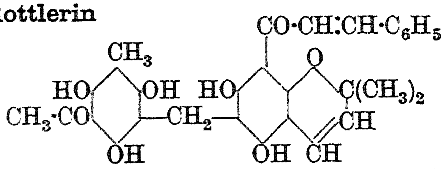
Occurs in resin from root of *Derris elliptica*, Benth. Needles or plates from Me_2CO.Aq. M.p. 163°. B.p. 210–20°/0·5 mm. Sol. ord. org. solvents. Sol. in EtOH reduces Fehling's and Tollen's. $[\alpha]_D^{29·5}$ − 225·2° in C_6H_6. Powerful contact insecticide and fish poison.

Oxime: (i) m.p. 249°. (ii) M.p. 230°.
Hydrazone: (i) m.p. 258° decomp. (ii) M.p. 229°.
Phenylhydrazone: (i) m.p. 255° (245°). (ii) M.p. 203°.

Takei, Miyajima, Ono, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1932, **19**, 1, (*Review and Bibl.*).
La Forge, Haller, Smith, *Chem. Reviews*, 1933, **12**, 181 (*Review*).
Cahn, Phipers, Boam, *J. Chem. Soc.*, 1938, 513.

Roteol.
See Isotubanol.

Rottlerin



$C_{30}H_{28}O_8$ MW, 516

Isolated from "kamala," an Indian colouring matter and anthelmintic drug, also from *Rottlera tinctoria*, Roxb. Yellowish brown cryst. from toluene or AcOEt. M.p. 212° (201–2°). O_3 or heating with dil. alkali ⟶ C_6H_5·CHO. H_2O_2 ⟶ cinnamic acid. On heating with EtOH or AcOH undergoes a series of complex rearrangements.
Di-Me ether: pale yellow leaflets from $CHCl_3$–MeOH or AcOEt–Me_2CO. M.p. 245–6° decomp.
Penta-Me ether: cryst. from pet. ether. M.p. 144°.
Penta-acetyl deriv.: plates from C_6H_6–EtOH or Me_2CO–EtOH. M.p. 214°.
Tetrahydro deriv.: pale yellow prisms from EtOH. M.p. 214°.

Dutt, *J. Chem. Soc.*, 1925, **127**, 2044.
Rojahn, *Chem. Abstracts*, 1937, **31**, 6816.
McGookin, Robertson, Tittensor, *J. Chem. Soc.*, 1939, 1579, 1587.
Robertson *et al.*, *J. Chem. Soc.*, 1948, 113.

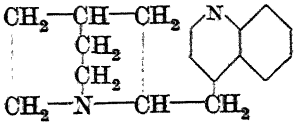
Rotundifoline

$C_{22}H_{26}O_5N_2$ MW, 398

Alkaloid from *Mitragyna rotundifolia*. Prisms from MeOH. M.p. 233–4°. Sol. $CHCl_3$. Mod. sol. Me_2CO, EtOH, C_6H_6. Spar. sol. Et_2O, AcOEt. $[\alpha]_D^{15}$ + 124° in $CHCl_3$. $FeCl_3$ ⟶ red col. Hyd. ⟶ rotundifolic acid, decomp. about 165°.

Barger, Dyer, Sargent, *J. Org. Chem.*, 1939, **4**, 418.

Ruban (2-*Quinuclidyl-4-quinolylmethane*)

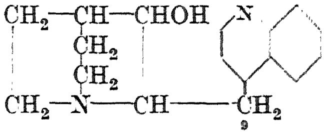


$C_{17}H_{20}N_2$ MW, 252

d-.
Oil. $[\alpha]_D^{16}$ + 80·5°.
l-.
Oil. $[\alpha]_D^{16}$ − 78·4°.

Rabe, Riza, *Ann.*, 1932, **496**, 151.
Rabe, *Ber.*, 1922, **55**, 523, 532.

Ruban-5-ol (5-*Hydroxyruban*)



$C_{17}H_{20}ON_2$ MW, 268

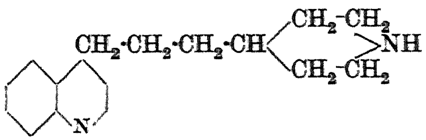
Needles. M.p. 198°. Sol. EtOH, C_6H_6. Spar. sol. Et_2O.
Picrate: yellow needles. M.p. 188–9°.

Clemo, Hoggarth, *J. Chem. Soc.*, 1939, 1241.

Ruban-9-ol (9-*Hydroxyruban*).
d : *d*-.
Cryst. from EtOH. M.p. 229·5–230° decomp. Sol. EtOH. Spar. sol. Et_2O. $[\alpha]_D^{16}$ + 132·5° in EtOH.
l-*Acid tartrate*: m.p. 186–7° decomp. $[\alpha]_D^{15}$ + 84·7° in H_2O.
d : *l*-.
Prisms from Et_2O. M.p. 118–19°. Sol. EtOH, Et_2O. $[\alpha]_D^{16}$ + 14·3° in EtOH.
Di-[dibenzyl]-tartrate: m.p. 189–91° decomp. $[\alpha]_D^{18}$ − 47·9° in MeOH.
l : *d*-.
M.p. 117–18°. $[\alpha]_D^{18}$ − 14·9° in EtOH.
Di-[dibenzyl]-tartrate: m.p. 189–91° decomp. $[\alpha]_D^{22}$ + 48·0° in MeOH.
l : *l*-.
M.p. 228·5–230·5° decomp. $[\alpha]_D^{15}$ − 131·8° in EtOH.
d-*Acid tartrate*: m.p. 186–8° decomp. $[\alpha]_D^{15}$ − 83·9° in H_2O.

Rabe, Riza, *Ann.*, 1932, **496**, 158.

Rubatoxan (1-[4-*Piperidyl*]-3-[4-*quinolyl*]-*propane*, 4-[ω-4-*piperidylpropyl*]-*quinoline*)



$C_{17}H_{22}N_2$ MW, 254

Yellowish liq. B.p. 185°/0·02 mm.
B,2HCl: cryst. from EtOH–AcOEt. M.p. 197°.
Chloroplatinate: orange cryst. from EtOH. M.p. above 360°.
Dipicrate: cryst. from EtOH. M.p. 203–5°.

Prelog, Seiwerth, Hahn, Cerkovnikov, *Ber.*, 1939, **72**, 1325.

Rubenol

$C_{20}H_{32}O_2$ MW, 304

Occurs in *Ceroplastes rubens*, Mask. Viscous liq. B.p. 188°/5 mm. Optically inactive. With Se ⟶ retene.

Diacetyl deriv.: b.p. 145–52°/6·5 mm.

Tetrahydro deriv.: b.p. 213°/7 mm.

Tetrabromo deriv.: m.p. 79–80°.

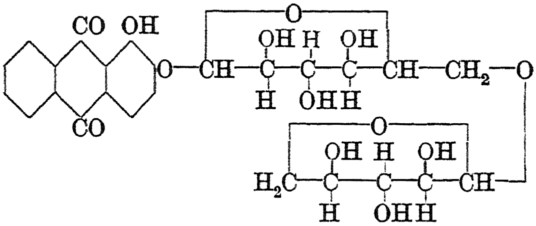
Nitrosate: m.p. 65–7°.

Dinitro deriv.: m.p. 80–1°.

Diphenylmethylurethane deriv.: m.p. 58–60°.

Kono, Maruyama, *Chem. Abstracts*, 1937, **31**, 5805; 1938, **32**, 6253.

Ruberythric Acid (*Alizarin primveroside*)



$C_{25}H_{26}O_{13}$ MW, 534

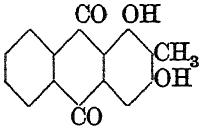
Occurs in *Rubia tinctorum*, Linn. Yellow prisms from H_2O. M.p. 258–60°. Spar. sol. H_2O, EtOH, Et_2O. Insol. C_6H_6. Sol. alkalis ⟶ red col. Hyd. ⟶ alizarin + glucose + xylose.

Octa-acetyl deriv.: m.p. 230°.

Penta-Me ether: $C_{30}H_{36}O_{13}$. MW, 604. Pale yellow needles from MeOH. M.p. 170–80°.

Richter, *J. Chem. Soc.*, 1936, 1701 (*Bibl.*).
Jones, Robertson, *J. Chem. Soc.*, 1933, 1167.
Zemplén, Müller, *Ber.*, 1929, **62**, 2107.
Glaser, Kahler, *Ber.*, 1927, **60**, 1349.

Rubiadin (*2-Methylpurpuroxanthin*, *2-methylxanthopurpurin*, 1 : *3-dihydroxy-2-methylanthraquinone*)



$C_{15}H_{10}O_4$ MW, 254

Occurs in root of *Rubia tinctorum*, Linn. (madder). Yellow plates from AcOH. M.p. 290°.

3-*Acetyl*: m.p. 191°.

Diacetyl: m.p. 225°.

3-β-*Glucoside*: occurs in *Rubia tinctorum*, Linn. $C_{21}H_{20}O_9$. MW, 416. Yellow needles from EtOH. M.p. 270–1°. *Tetra-acetyl*: m.p. 230°.

1 : 3-*Di*-β-*glucoside*: m.p. 248°.

Primeveroside: occurs in *Galium verum*, Linn. $C_{26}H_{28}O_{13}$. MW, 548. Yellow plates. M.p. 248–50°. Sol. hot H_2O. Hyd. ⟶ rubiadin-3-glucoside + xylose.

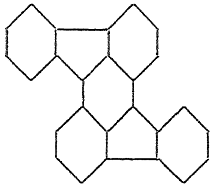
1-*Me ether*: $C_{16}H_{12}O_4$. MW, 268. Yellow needles from EtOH. M.p. 291°. *Acetyl*: m.p. 174°.

3-*Me ether*: yellow plates from AcOH. M.p. 186°. *Acetyl*: m.p. 200°.

Di-Me ether: $C_{17}H_{14}O_4$. MW, 282. Yellow needles from MeOH. M.p. 158°.

Jones, Robertson, *J. Chem. Soc.*, 1930, 1699.
Kusaka, *Chem. Zentr.*, 1935, II, 3381.
Mitter, Biswas, *J. Indian Chem. Soc.*, 1930, **7**, 839.
Hill, Richter, *J. Chem. Soc.*, 1936, 1714.

Rubicene



$C_{26}H_{14}$ MW, 326

Red needles from $PhNO_2$. M.p. 306°. Sol. hot $PhNO_2$. Spar. sol. C_6H_6. Insol. EtOH, Et_2O, pet. ether. Dil. sols. show intense yellow fluor.

Scholl, Meyer, *Ber.*, 1932, **65**, 926.
Eckert, *J. prakt. Chem.*, 1929, **121**, 278.
v. Braun, *Ber.*, 1934, **67**, 217.

Rubijervine

$C_{27}H_{43}O_2N$ MW, 413

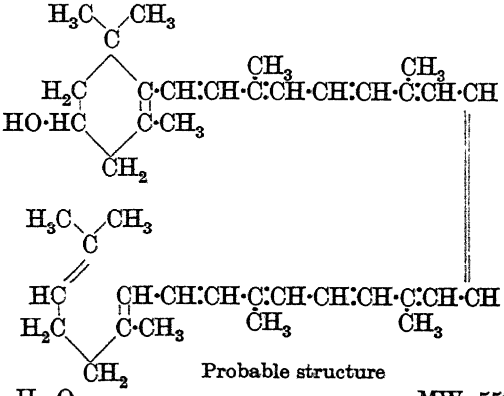
Alkaloid from *Veratrum album* and *Veratrum viride*. Cryst. + $1H_2O$ from EtOH or Me_2CO. M.p. 242°. $[\alpha]_D$ + 19° in EtOH. Conc. H_2SO_4 ⟶ red col.

B,HI: needles from Me_2CO-Et_2O. M.p. 261–2° decomp.

p-*Bromobenzoyl*: cryst. from EtOH-C_6H_6. M.p. 254–6° decomp.

Poethke, *Arch. Pharm.*, 1938, **276**, 170.
Jacobs, Craig, *J. Biol. Chem.*, 1943, **148**, 41; 1943, **149**, 451; 1945, **159**, 617; 1945, **160**, 555.

Rubixanthin



Probable structure

$C_{40}H_{56}O$ MW, 552

Pigment of ripe fruit of *Rosa rubinosa*. Dark red needles from C_6H_6–MeOH. M.p. 160°. Absorption maxima in CS_2 at 533, 494 and 461 mμ.

Kuhn, Grundmann, *Ber.*, 1934, **67**, 339.

Rubradinine (*Mitraphyline*)

$C_{24}H_{28}O_4N_2$ MW, 408

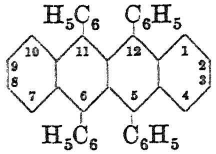
Alkaloid from *Adina rubrostipulata*, K. Schum. Needles from 95% EtOH. M.p. 306°. $[\alpha]_D$ − 22·3° in $CHCl_3$.

$B,H_2SO_4,5H_2O$: m.p. 245°.

Picrate: m.p. 166°.

Denis, *Chem. Abstracts*, 1937, **31**, 3928; *Brit. Chem. Abstracts*, 1940, A II, 262.

Rubrene (5 : 6 : 11 : 12-*Tetraphenyl*-1 : 2-*benzanthracene*



$C_{42}H_{28}$ MW, 532

Red cryst. M.p. 331°. Sol. C_6H_6, CS_2. Sols. are orange when conc., pink with yellow fluor. when dil.

Koblitz, Witmeyer, *Ber.*, 1936, **69**, 1806.
Robin, *Compt. rend.*, 1929, **189**, 337.
Allen, Gilman, *J. Am. Chem. Soc.*, 1936, **58**, 937.
Bergmann, Herlinger, *J. Chem. Physics*, 1936, **4**, 532.
Dufraisse, *Bull. soc. chim.*, 1930, **47**, 216.

Rubreserine

$C_{13}H_{16}O_2N_2$ MW, 232

Oxidation product of eseroline. Red needles $+1H_2O$ from H_2O. M.p. anhyd. 152°. Sol. H_2O, EtOH, $CHCl_3$, hot C_6H_6. Insol. Et_2O, pet. ether.

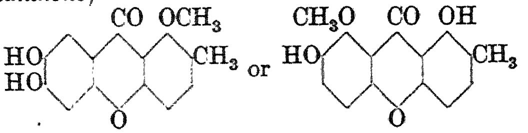
B,HCl: red cryst. $+ 1H_2O$. M.p. 185° decomp.

$B,HAuCl_4$: decomp. at 190–5°.

Picrate: decomp. at 198°.

Salway, *J. Chem. Soc.*, 1912, **101**, 984.

Rubrofusarin (*Dihydroxymethoxymethylxanthone*)



$C_{15}H_{12}O_5$ MW, 272

Pigment of *Fusarium culmorum* Sacc., and related forms. Orange red needles from pet. ether, C_6H_6 or EtOH. M.p. 210–11°. Insol. H_2O. Spar. sol. aq. alkalis. $FeCl_3$ ⟶ greenish brown col.

Monoacetyl deriv.: golden yellow hexagonal prisms from C_6H_6 or AcOH. M.p. 211°.

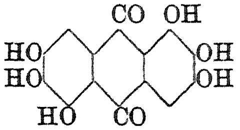
Diacetyl deriv.: prac. colourless rods. M.p. 260°.

Me ether: m.p. 203–4°.

Di-Me ether: m.p. 187–8°. *Ferrichloride*: maroon prisms from AcOH. M.p. 183–4° decomp.

Ashley, Hobbs, Raistrick, *Biochem. J.*, 1937, **31**, 385.
Mull, Nord, *Arch. Biochem.*, 1944, **4**, 419.

Rufigallic Acid (*Rufigallol*, 1 : 2 : 3 : 5 : 6 : 7-*hexahydroxyanthraquinone*)



$C_{14}H_8O_8$ MW, 304

Red cryst. Sublimes with decomp. on heating. Sol. EtOH, Et_2O. Sol. alkalis ⟶ violet col. Heat of comb. C_p 1252·4 Cal.

Tetra-Me ether: $C_{18}H_{16}O_8$. MW, 360. Yellow leaflets from AcOEt. M.p. 235–7°. *Diacetyl deriv.*: m.p. 262°. *Monobenzoyl deriv.*: m.p. 190–205°.

Penta-Me ether: $C_{19}H_{18}O_8$. MW, 374. Yellow needles from AcOEt. M.p. 192–4°.

Hexa-Me ether: $C_{20}H_{20}O_8$. MW, 388. Yellow needles. M.p. 245° (240°).

Tri-Et ether: $C_{20}H_{20}O_8$. MW, 388. Orange-red needles from EtOH. M.p. 195°.

Tetra-Et ether: $C_{22}H_{24}O_8$. MW, 416. Red needles from EtOH. M.p. above 180°. *Diacetyl deriv.*: m.p. 230–5°.

Hexa-Et ether: $C_{26}H_{32}O_8$. MW, 472. Orange-yellow needles from EtOH.Aq. M.p. above 140°.

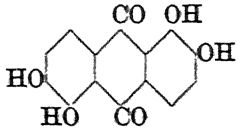
Hexa-acetyl: m.p. 282–3°.

Segui, *Chem. Abstracts*, 1934, **28**, 7257.
Fischer, Gross, *J. prakt. Chem.*, 1911, **84**, 369.
Schering, D.R.P., 151,724, (*Chem. Zentr.*, 1904, I, 1586).

Rufigallol.

See Rufigallic Acid.

Rufiopin (1 : 2 : 5 : 6-*Tetrahydroxyanthraquinone*)



$C_{14}H_8O_6$ MW, 272

Orange-red needles from Py. M.p. 340° (316–18°). Sol. hot H_2O. Mod. sol. EtOH, AcOH. Spar. sol. Et_2O, C_6H_6, $CHCl_3$.

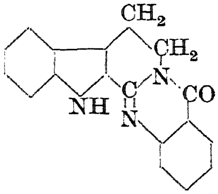
1 : 2 : 6-*Tri-Me ether*: $C_{17}H_{14}O_6$. MW, 314. Orange-red needles from AcOH. M.p. 245–6°.

Tetra-acetyl: m.p. 260–3° decomp. (238°, 260–75° decomp.).

Heller, *Z. angew. Chem.*, 1929, **42**, 170.
Puntambeker, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 487.
Marshall, *J. Chem. Soc.*, 1937, 254.

Rufol.
See 1 : 5-Dihydroxyanthracene.

Rutæcarpine



$C_{18}H_{13}ON_3$ MW, 287

Alkaloid from fruit of *Evodia rutæcarpa*. Yellow needles from EtOH. M.p. 256°. Conc. H_2SO_4 ⟶ yellow col.

Schöpf, Steuer, *Ann.*, 1947, **558**, 124.

Ruthenium di-cyclopentadienyl.
See Ruthenocene.

Ruthenocene (*Ruthenium di-cyclopentadienyl*)

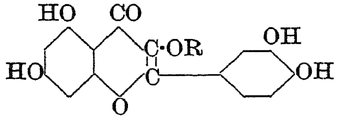
$(C_5H_5)_2Ru$

$C_{10}H_{10}Ru$ MW, 232

Transparent, plate-like cryst. from CCl_4. M.p. 195·5°.

Wilkinson, *J. Am. Chem. Soc.*, 1952, **74**, 6146, 6149.

Rutin (*Eldrin, violaquercitrin, osyritrin, myrticolorin, quercetin rutinoside*)



R = Rutinose

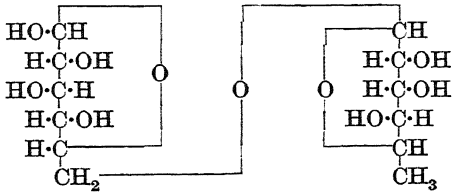
$C_{27}H_{30}O_{16}$ MW, 610

Occurs in tomato stems, tobacco leaves, rue leaves and many flowers. Yellow needles $+ 2H_2O$ from H_2O. Softens below 188°, not completely melted at 190°. Sol. hot EtOH. Insol. Et_2O, $CHCl_3$, C_6H_6, CS_2. $FeCl_3$ ⟶ intense green col.

Deca-Me deriv.: $[\alpha]_D^{19}$ − 33° in EtOH.

Charaux, *Bull. soc. chim. biol.*, 1924, **6**, 641.
Sanso, Lloyd, *J. Biol. Chem.*, 1924, **58**, 737.
Perkin, *J. Chem. Soc.*, 1900, **97**, 1788.
Zemplén, Gerecs, *Ber.*, 1935, **68**, 1318.
Zemplén, Tettamanti, *Ber.*, 1938, **71**, 2511.

Rutinose



$C_{12}H_{22}O_{10}$ MW, 326

Hygroscopic powder. Softens at 140°, m.p. 189–92° decomp. Sol. H_2O, EtOH. Insol. Et_2O. $[\alpha]_D^{10}$ + 3·24° ⟶ − 0·81° in H_2O, $[\alpha]_D^{20}$ − 10·0° in EtOH. Reduces Fehling's.

Hepta-acetyl deriv.: m.p. 168–9°. $[\alpha]_D$ − 28·84° in $CHCl_3$. *Me ether*: m.p. 139·5–140°. $[\alpha]_D$ − 45·19°.

α-Acetobromo deriv.: m.p. 127–8°. $[\alpha]_D^{16}$ + 89° in $CHCl_3$.

Hepta-Me ether: oil. $[\alpha]_D^{18}$ + 2·6° in H_2O.

Zemplén, Gerecs, *Ber.*, 1935, **68**, 1319.
Charaux, *Compt. rend.*, 1924, **178**, 1312.
Zemplén, Gerecs, *Ber.*, 1937, **70**, 1098.

Rutylidene.
See Nonylacetylene.

Ryanodin

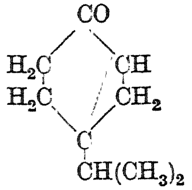
$C_{25}H_{35}O_9N$ MW, 493

Occurs in root of *Ryania speciosa*, Vahl. Cryst. from Et_2O. M.p. 219–20°. $[\alpha]_D^{25}$ + 26° in MeOH. Sol. H_2O, EtOH, Me_2CO, Et_2O, $CHCl_3$. Spar. sol. C_6H_6. Insol. pet. ether. Neutral. Forms no salts. Conc. H_2SO_4 ⟶ red col. changing to greenish-brown. Conc. HNO_3 ⟶ purple-red col. Contains pyrrole system.

Rogers *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 3086.
Kelly, Whittingham, Wiesner, *Canadian Journal of Chemistry*, 1951, **29**, 905; *Chemistry and Industry*, 1952, 857.

S

Sabinaketone



$C_9H_{14}O$ MW, 138

l-.

Prisms from H_2O. M.p. 17°. B.p. 218–19°. D^{20} 0·9555. n_D^{20} 1·4700. $[\alpha]_D^{15\cdot6}$ − 19·8°, $[\alpha]_D^{15}$ − 34·2° in EtOH. Spar. sol. H_2O.

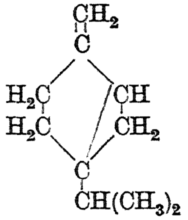
Semicarbazone: cryst. from MeOH. M.p. 135–7°. $[\alpha]_D^{15\cdot6}$ + 137·5° in MeOH.

Dinitrophenylhydrazone: orange needles from MeOH–$CHCl_3$. M.p. 124°. $[\alpha]_D$ + 135·2° in $CHCl_3$.

Schmidt, *Z. angew. Chem.*, 1929, **42**, 127.
Wallach, *Ann.*, 1908, **359**, 266.
Short, Read, *J. Chem. Soc.*, 1939, 1415.

Sabinane.
See Thujane.

Sabinene



$C_{10}H_{16}$ MW, 136

d-.

Constituent of various essential oils, e.g. *Juniperus sabina*. B.p. 163–5°, 66°/30 mm. D^{20} 0·842. n_D^{20} 1·4678. $[\alpha]_D$ + 80·17° (89·07°).

l-.

B.p. 162–6°. D^{20} 0·8468. $[\alpha]_D^{15}$ − 42·5°.

Hydrate: methylsabinaketol. Cryst. resembling terpineol in odour. M.p. 38–9°. B.p. 195–201° part. decomp. $[\alpha]_D^{15}$ + 53·67° in Et_2O.

Semmler, *Ber.*, 1900, **33**, 1463.
Wallach, *Ann.*, 1907, **357**, 64, 77.

Sabinic Acid (11-*Hydroxylauric acid*)

$HO{\cdot}CH_2{\cdot}[CH_2]_9{\cdot}CH_2{\cdot}COOH$

$C_{12}H_{24}O_3$ MW, 216

Cryst. from C_6H_6 or AcOEt, m.p. 84°: needles from MeOH.Aq, m.p. 78–9°. Sol. EtOH.

Me ester: $C_{13}H_{26}O_3$. MW, 230. M.p. 44·5° (34–5°). B.p. 160°/7 mm. (164–6°/3 mm.).

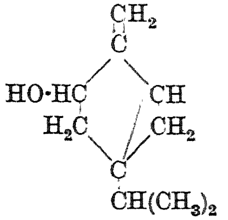
Phenylurethane: m.p. 64–5°.

Anilide: prisms from pet. ether. M.p. 87°.

Acetyl: m.p. 49–49·2° (43°). B.p. 202–3°.

Chuit, Hausser, *Helv. Chim. Acta*, 1929, **12**, 477.
Bhattacharya, Saletore, Simonsen, *J. Chem. Soc.*, 1928, 2679.
Lycan, Adams, *J. Am. Chem. Soc.*, 1929, **51**, 628.

Sabinol



$C_{10}H_{16}O$ MW, 152

Constituent of oils of *Juniperus sabina* and *Sabina officinalis*. B.p. 208°, 90°/11 mm. D_4^{25} 0·9461. n_D^{25} 1·4871. $[\alpha]_D^{19}$ + 4°.

Acetyl: b.p. 225°, 81–2°/3 mm. D^{15} 0·972.

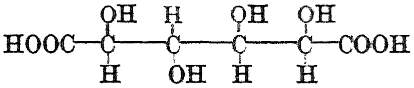
Acid phthalate: needles from pet. ether. M.p. 101–2°. $[\alpha]_D^{17}$ −17·5° in MeOH, + 10° in $CHCl_3$.

p-*Nitrobenzoyl*: m.p. 76°. $[\alpha]_D^{13}$ + 10° in $CHCl_3$.

3 : 5-*Dinitrobenzoyl*: yellow cryst. M.p. 109°. $[\alpha]_D^{13}$ + 22·5° in $CHCl_3$.

Fromm, *Ber.*, 1898, **31**, 2025.
Short, Read, *J. Chem. Soc.*, 1939, 1040.

Saccharic Acid (*Tetrahydroxyadipic acid, glucosaccharic acid*)



$C_6H_{10}O_8$ MW, 210

d-.

Needles from 95% EtOH. M.p. 125–6°. $[\alpha]_D^{19}$ + 6·86° ⟶ + 20·60° in H_2O. Sol. H_2O, EtOH. Spar. sol. Et_2O. *k* (first) = 1·0 × 10^{-5} at 25°. HNO_3 ⟶ *d*-tartaric, racemic, and oxalic acids. $KMnO_4$ ⟶ *d*-tartaric and oxalic acids.

Cinchonine salt: $2C_{19}H_{22}ON_2,C_6H_{10}O_8$. Cryst. from H_2O. Decomp. about 190°. $[\alpha]_D^{20}$ + 149·1° in H_2O.

Quinine salt: $2C_{20}H_{24}O_2N_2,C_6H_{10}O_8$. Needles. M.p. 174°.

Di-Me ester: $C_8H_{14}O_8$. MW, 238. *Tetra-Me ether*: prisms from Et_2O. M.p. 77–8° (68°). B.p. 150°/1 mm. $[\alpha]_D^{18}$ + 8·88° ⟶ + 10·26° in H_2O. Sol. H_2O, Et_2O, $CHCl_3$.

Di-Et ester: $C_{10}H_{18}O_8$. MW, 266. Cryst. mass. Sol. H_2O, EtOH. Spar. sol. Et_2O.

Tetra-acetyl: plates from EtOH or prisms from Et_2O. M.p. 61°. Sol. hot EtOH, Et_2O. Insol. H_2O.

Amide: $C_6H_{11}O_7N$. MW, 209. Needles. M.p. 135° decomp. $[\alpha]_D^{19}$ + 22·5° in H_2O. Sol. hot H_2O. Spar. sol. EtOH.

Diamide: $C_6H_{12}O_6N_2$. MW, 208. Cryst. from EtOH. M.p. 172–3°. $[\alpha]_D^{20}$ + 13·3° in H_2O. 2 : 5-*Di-Me ether*: m.p. 183–5° decomp. $[\alpha]_D^{20}$ − 55° in H_2O. *Tetra-Me ether*: plates from H_2O. M.p. 237–9°. $[\alpha]_D^{18}$ + 12·22° in H_2O.

1 : 4-*Lactone*: cryst. + $1H_2O$. M.p. 90°.

3 : 6-*Lactone*: $C_6H_8O_7$. MW, 192. Cryst. M.p. 130–2°. Sol. H_2O. $[\alpha]_D^{25}$ + 40·8° ⟶ + 22·7° in H_2O. *Me ester*: m.p. 113–14°. $[\alpha]_D$ + 29° in H_2O. *Et ester*: m.p. 126–7°.

γγ-Dilactone: 2 : 5-*diacetyl deriv.*: m.p. 190–2°. $[\alpha]_D$ + 155°.

Diphenylhydrazide: yellow plates. Decomp. at 210°. Insol. H_2O, EtOH, Et_2O.

l-.

Free acid not isolated.

KH salt: needles or prisms from hot H_2O. Mod. sol. H_2O.

Diphenylhydrazide: yellow plates. M.p. 213–14° decomp.

dl-.

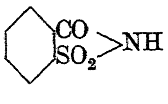
Syrup.

KH salt: needles. Mod. sol. H_2O.

Diphenylhydrazide: laminæ. M.p. 209–10° decomp.

Sohst, Tollens, *Ann.*, 1888, **245**, 1.
Henneberg, Tollens, *Ann.*, 1896, **292**, 40.
Fischer, *Ber.*, 1890, **23**, 2621.
Fischer, Stahel, *Ber.*, 1891, **24**, 534.
Kiliani, *Ber.*, 1925, **58**, 2344.
Rehorst, *Ber.*, 1928, **61**, 163.
Haworth, Loach, Long, *J. Chem. Soc.*, 1927, 3154.
Karrer, Peyer, *Helv. Chim. Acta*, 1922, **5**, 577.
Bergmann, *Ber.*, 1921, **54**, 1380, 2653.
Hudson, Komatsu, *J. Am. Chem. Soc.*, 1919, **41**, 1147.
Schmidt, Günthert, *Ber.*, 1938, **71**, 493.
Reeves, *J. Am. Chem. Soc.*, 1939, **61**, 664.
Mayumi, Wada, *Chem. Abstracts*, 1949, **43**, 7904.

Saccharin (*Benzoic sulphimide, o-sulphobenzoic imide*)



$C_7H_5O_3NS$ MW, 183

Prisms from EtOH, leaflets from H_2O. M.p. 224° decomp. Sol. 250 parts cold H_2O, 40 parts cold EtOH, 221 parts hot C_6H_6. Sublimes in vacuo. Forms salts with NH_3 and org. bases. All salts intensely sweet. Hot alkalis or hot conc. HCl ⟶ *o*-sulphobenzoic acid.

Oxime: m.p. 208–10° decomp. *Acetate*: m.p. 225° decomp. *Benzoate*: m.p. 250° decomp.

Semicarbazone: silvery cryst. M.p. 230–5°.

N-*Me*: $C_8H_7O_3NS$. MW, 197. Needles from H_2O. M.p. 131–2°. Sol. EtOH, Et_2O. Spar. sol cold H_2O.

N-*Et*: $C_9H_9O_3NS$. MW, 211. Needles from H_2O. M.p. 93–4°. Very sol. EtOH, Et_2O. Mod. sol. hot H_2O.

Zaikov, Sokolov, *Chem. Abstracts*, 1928, **22**, 2933.
Brackett, Hayes, *Am. Chem. J.*, 1887, **9**, 405.
Zil'berg, *Chem. Abstracts*, 1935, **29**, 1794.
Roost, *Chem. Abstracts*, 1928, **22**, 4114.
Orelup, U.S.P. 1,601,505, (*Chem. Zentr.*, 1927, II, 2115).

Saccharose.

See Sucrose.

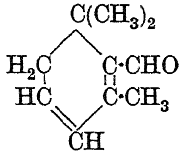
S-Acid.

See 1-Amino-8-naphthol-4-sulphonic Acid.

2S-Acid.

See 1-Amino-8-naphthol-2 : 4-disulphonic Acid.

Safranal



$C_{10}H_{14}O$ MW, 150

Hydrolysis product of picrocrocin. B.p. 172°, 70°/1 mm. D_4^{19} 0·9734. n_D^{19} 1·5281.

Oxime: prisms from pet. ether. M.p. 65°.

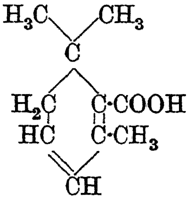
Semicarbazone: cryst. from MeOH. M.p. 175°.

Thiosemicarbazone: cryst. from C_6H_6. M.p. 199–200°.

2 : 4-*Dinitrophenylhydrazone*: red cryst. from MeOH. M.p. 186°.

Kuhn, Wendt, *Ber.*, 1936, **69**, 1549.
Kuhn, Winterstein, *Ber.*, 1934, **67**, 354.

Safranic Acid (2 : 6 : 6-*Trimethyl*-1 : 3-*cyclohexadiene*-1-*carboxylic acid*)



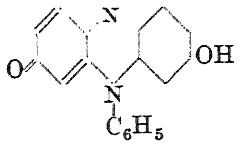
$C_{10}H_{14}O_2$ MW, 166

Prisms from pet. ether. M.p. 63–4°.

p-*Bromophenacyl ester*: m.p. 102°.

Gehard, Wenat, *Ber.*, 1941, **74**, 1242.

Safranol



$C_{18}H_{12}O_2N_2$ MW, 288

Needles. Does not melt below 330°. Very spar. sol. Et_2O. Insol. H_2O, EtOH, AcOH. Sol. alkalis.

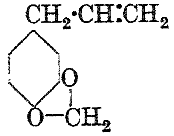
Me ether: $C_{19}H_{14}O_2N_2$. MW, 302. M.p. 266°.

Et ether: $C_{20}H_{16}O_2N_2$. MW, 316. Bronze prisms from EtOH. M.p. 265° decomp.

Acetyl: red prisms with greenish lustre. M.p. 265–8°.

Jaubert, *Ber.*, 1895, **28**, 273.

Safrol (3 : 4-*Methylenedioxyallylbenzene*)



$C_{10}H_{10}O_2$ MW, 162

Constituent of several essential oils, e.g. *Sassafras officinalis*. Monoclinic prisms. Setting-point 11·2°. B.p. 231·5–232°, 104–5°/6 mm. D_4^{20} 1·100. n_D^{20} 1·5383. Sol. EtOH, Et_2O. Insol. H_2O.

Picrate: red cryst. from EtOH. M.p. 75°.

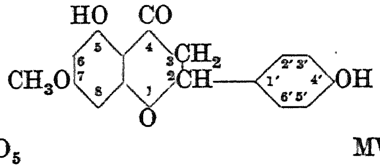
Maleic anhydride adduct: yellow cryst. from toluene. M.p. 257°.

Perkin, Trikojus, *J. Chem. Soc.*, 1927, 1663.
Berta, *Compt. rend.*, 1941, **213**, 873.

Saiodin.

See under Iodobehenic Acid.

Sakuranetin (*Naringenin 7-methyl ether, 5:4′-dihydroxy-7-methoxyflavanone*)



$C_{16}H_{14}O_5$ MW, 286

Needles from EtOH.Aq., dry Et_2O, or C_6H_6. M.p. 152–4°. Cryst. from H_2O with $3H_2O$. M.p. 222°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, AcOEt, Py. Fuming $HNO_3 \longrightarrow$ deep indigo-blue col. $\longrightarrow$ violet on standing. Alkalis $\longrightarrow$ intense yellow sols. decomp. by CO_2 and weak acids. Mg + alc. HCl $\longrightarrow$ purplish-red col. KOH fusion $\longrightarrow$ phloroglucinol + *p*-hydroxybenzoic acid. Tasteless.

4′-Me ether: *see under* Isosakuranetin.

Di-Me ether: *see under* Isosakuranetin.

Oxime: needles + $2H_2O$ from EtOH.Aq., m.p. 120°: needles from AcOH, m.p. 195–6° decomp.

5-Glucoside: *see* Sakuranin.

Diacetyl: m.p. 97°.

Shinoda, Sato, *Chem. Zentr.*, 1929, I, 244.
Asahina, Shinoda, Inubuse, *Chem. Zentr.*, 1928, I, 1672.

Sakuranin (*Sakuranetin-5-glucoside*)

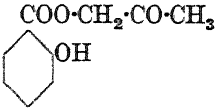
$C_{22}H_{24}O_{10}$ MW, 448

Constituent of rind of *Prunus yeodensis*. Needles from EtOH or AcOEt. M.p. 212–14°. Very sol. 60% EtOH, Py. Less sol. EtOH. Insol. H_2O, Et_2O. $[\alpha]_D^{23}$ − 123·2° in anhydrous Me_2CO. Alkalis $\longrightarrow$ intense yellow sols. Conc. $H_2SO_4 \longrightarrow$ intense brown col. $\longrightarrow$ yellow on adding more acid. Alc. $FeCl_3 \longrightarrow$ yellow col.

Oxime: needles from EtOH. M.p. 110°. Conc. $HNO_3 \longrightarrow$ green col.

Asahina, *Chem. Zentr.*, 1908, II, 253.
Asahina, Shinoda, Inubuse, *Chem. Zentr.*, 1928, I, 1672.
Zemplén, Bognár, Mester, *Ber.*, 1942, **75**, 1432.

Salacetol (*Acetol salicylate, hydroxyacetone salicylate, β-ketopropyl salicylate*)



$C_{10}H_{10}O_4$ MW, 194

Needles from EtOH. M.p. 71°. Sol. Et_2O, C_6H_6, hot EtOH. Spar. sol. ligroin. Very spar. sol. hot H_2O. Triboluminescent.

Fritsch, D.R.P., 70,054.

Salazine (*Salicylaldazine, 2 : 2′-dihydroxybenzaldazine*)

$C_{14}H_{12}O_2N_2$ MW, 240

Yellow needles or leaflets from EtOH. M.p. 214°. Sol. $CHCl_3$, C_6H_6, alkalis. Mod. sol. EtOH. Insol. H_2O. Sublimes.

Di-Me ether: $C_{16}H_{16}O_2N_2$. MW, 268. Yellow needles from EtOH. M.p. 178° (141°).

Di-Et ether: $C_{18}H_{20}O_2N_2$. MW, 296. Yellow cryst. from EtOH. M.p. 136° (130°).

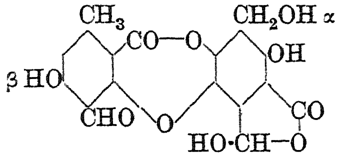
Dibenzyl ether: yellow plates from C_6H_6. M.p. 158°.

Diacetyl: plates from $CHCl_3$. M.p. 191°.

Dibenzoyl: yellow needles from C_6H_6. M.p. 188–9°.

Widman, *Ber.*, 1919, **52**, 1658.
Borsche, *Ber.*, 1901, **34**, 4299; 1921, **54**, 668.
Curtius, Jay, *J. prakt. Chem.*, 1889, **39**, 48.

Salazinic Acid (*Saxatilic acid*)



Probable structure

$C_{18}H_{12}O_{10}$ MW, 388

Isolated from *Parmelia conspersa*, Ach. Needles or plates from Me_2CO. M.p. 270° (decomp. at 260°). Sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. EtOH, Me_2CO. $FeCl_3 \longrightarrow$ brownish-red col.

α-Me ether: $C_{19}H_{14}O_{10}$. MW, 402. Needles from 80% Me_2CO. M.p. 210° decomp. Sol. EtOH, Me_2CO, AcOH. Spar. sol. Et_2O. Insol. C_6H_6, pet. ether. *Dianil*: yellow prisms from Me_2CO. M.p. 169° decomp.

β-Me ether: needles from 80% Me_2CO. M.p. 250–2° decomp. Sol. EtOH, Me_2CO, AcOH. Insol. Et_2O, C_6H_6, pet. ether. *Penta-acetyl*: plates from Me_2CO. M.p. 228° decomp. *Dianil*: yellow prisms from Me_2CO. M.p. 231° decomp.

α-Acetyl: needles from AcOH. M.p. 275–6° decomp. Sol. EtOH, Me_2CO.

Tetra-acetyl: m.p. 205–6°.

Hexa-acetyl: needles from Me_2CO. M.p. 178°.

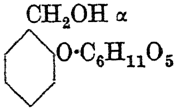
Di-phenylhydrazone: yellow prisms. Decomp. at 295°.

Dianil: M.p. 240°. Decomp. at 280°.

Asahina, Tukamoto, *Ber.*, 1934, **67**, 965.
Koller, Kutzelnigg, *Monatsh.*, 1934, **65**, 92.
Asahina, Asano, *Ber.*, 1933, **66**, 895.
Fuzikawa, Ishiguro, *Chem. Zentr.*, 1937, I, 2996.

Saleripol.
See Gentisyl Alcohol.

Salicin (*Saligenin glucoside*)



$C_{13}H_{18}O_7$ MW, 286

Glucoside of poplar and willow bark. Needles from H_2O. M.p. 204·7–208·7°. Sol. 24 parts H_2O at 25°. $[\alpha]_D^{20}$ − 62·56° in H_2O. Hydrolysis by emulsin $\longrightarrow$ glucose + saligenin. H_2SO_4 $\longrightarrow$ purple col. Possesses burning taste.

Penta-Me ether: $C_{18}H_{28}O_7$. MW, 356. Needles from pet. ether. M.p. 62–4°. $[\alpha]_D$ − 52·1° in MeOH.

Tetra-acetyl: needles from H_2O. M.p. 130° (126°). Insol. pet. ether. *α-Me ether*: plates from EtOH. M.p. 142°. *α-Allyl ether*: needles from EtOH. M.p. 139·5°. *α-Benzyl ether*: plates from EtOH or pet. ether. M.p. 94·5–95°. *α-Phenyl ether*: yellow needles from EtOH. M.p. 161°.

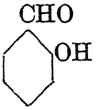
Penta-acetyl: cryst. from EtOH. M.p. 130°. $[\alpha]_D^{23\cdot5}$ − 18·5° in $CHCl_3$.

Benzoyl: *see* Populin.

Kunz, *J. Am. Chem. Soc.*, 1926, **48**, 262.
Zemplén, Braun, *Ber.*, 1925, **58**, 1406.
Irvine, Rose, *J. Chem. Soc.*, 1906, **89**, 814.

Salicyl Alcohol.
See Saligenin.

Salicylaldehyde (o-*Hydroxybenzaldehyde*)



$C_7H_6O_2$ MW, 122

Oil. F.p. − 7°. B.p. 197°, 93°/25 mm., 86°/18 mm. D_4^{20} 1·1674. n_D^{20} 1·574. Heat of comb. C_p 807·3 Cal. Volatile in steam. Does not reduce Fehling's. $H_2O_2 \longrightarrow$ 2 : 3-dihydroxybenzaldehyde. KOH $\longrightarrow$ salicylic acid. NaHg $\longrightarrow$ saligenin. $FeCl_3 \longrightarrow$ violet col. $H_2SO_4 \longrightarrow$ orange col.

Me ether: *see* o-Methoxybenzaldehyde.

Et ether: o-ethoxybenzaldehyde. $C_9H_{10}O_2$. MW, 150. M.p. 20–2°. B.p. 247–9°, 143–7°/25 mm. *Diacetate*: prisms. M.p. 88–9°. *Oxime*: prisms from pet. ether. M.p. 57–9°. *Semicarbazone*: needles from EtOH. M.p. 219°.

Allyl ether: $C_{10}H_{10}O_2$. MW, 162. B.p. 130°/10 mm.

Benzyl ether: $C_{14}H_{12}O_2$. MW, 212. Prisms from EtOH. M.p. 46°. *Oxime*: plates from pet. ether. M.p. 71·5°.

Acetyl: needles from pet. ether. M.p. 38–9°. B.p. 142°/18 mm. *Semicarbazone*: powder from Me_2CO. M.p. 167°.

Triacetate: o-acetoxybenzylidene diacetate. Prisms from EtOH. M.p. 107°.

Benzoyl: b.p. above 300°.

p-*Nitrobenzoyl*: yellow plates. M.p. 128°.

Oxime: prisms from C_6H_6–pet. ether. M.p. 63° (57°). *Me ether*: (>N·OCH_3). Prisms. M.p. 28°. B.p. 107°/14 mm. *Acetate*: (>N·OCO·CH_3). Needles from pet. ether. M.p. 75° (69°).

Semioxamazone: needles from EtOH.Aq. M.p. 255°.

Semicarbazone: needles from EtOH. M.p. 230°.

Phenylsemicarbazone: needles. M.p. 198–200°.

o-*Tolylsemicarbazone*: needles. M.p. 204·5°.

p-*Tolylsemicarbazone*: needles. M.p. 238–9°.

1-*Naphthylsemicarbazone*: m.p. 213–14°.

2-*Naphthylsemicarbazone*: m.p. 202–3°.

Thiosemicarbazone: m.p. 231° (sinters at 215°).

Phenylthiosemicarbazone: needles or plates. M.p. 183°.

Hydrazone: plates from EtOH. M.p. 96°.

Phenylhydrazone: needles or plates. M.p. 142–3°. Sensitive to light. *Dibenzoyl*: m.p. 170–1°.

2 : 4 : 6-*Trichlorophenylhydrazone*: needles from EtOH or AcOH. M.p. 98·5–99·5°.

o-*Bromophenylhydrazone*: yellow cryst. M.p. 176–8°.

p-*Bromophenylhydrazone*: yellow plates. M.p. 175·5°.

p-*Iodophenylhydrazone*: m.p. 214–15°.

2 : 4 : 6-*Tribromophenylhydrazone*: needles from EtOH. M.p. 100°.

o-*Nitrophenylhydrazone*: m.p. 193°.

m-*Nitrophenylhydrazone*: m.p. 197°.

p-*Nitrophenylhydrazone*: reddish-brown prisms. M.p. 227°.

2 : 4-*Dinitrophenylhydrazone*: purplish-red needles from EtOH. M.p. 248–52° decomp.

o-*Tolylhydrazone*: m.p. 111–12°.

1-*Naphthylhydrazone*: yellow plates. M.p. 134°.

2-*Naphthylhydrazone*: yellow needles. M.p. 187°.

Benzoylhydrazone: needles from EtOH.Aq. M.p. 182°.

1-*Naphthoylhydrazone*: needles. M.p. 235°.

Phenylurethane: m.p. 133°.

p-*Toluenesulphonyl*: cryst. from MeOH. M.p. 63–4°.

Glucoside: *see* Helicin.

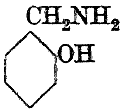
Azine: *see* Salazine.

Anil: *see* Salicylideneaniline.

Dimethone: m.p. 205–6°.

Claisen, Eisleb, *Ann.*, 1913, **401**, 95.
Brady, Dunn, *J. Chem. Soc.*, 1914, **105**, 825.
Kawada, Yosida, *Chem. Abstracts*, 1930, **24**, 1040.
Copisarow, *J. Chem. Soc.*, 1929, 588.
B.D.C., B.P. 232,392, (*Chem. Zentr.*, 1925, II, 1226).
Weiss, Donns, U.S.P., 1,380,277, (*Chem. Zentr.*, 1921, IV, 587).
Duff, *J. Chem. Soc.*, 1941, 547.

Salicylamine (o-*Hydroxybenzylamine*, ω-*amino*-o-*cresol*)



C_7H_9ON MW, 123

Cryst. from EtOH–pet. ether. M.p. 129° after softening at 126°. Sol. H_2O, EtOH, Et_2O. Insol. pet. ether. Sublimes readily. $FeCl_3$ ⟶ violet-blue col. ⟶ red on heating.

Me ether: o-methoxybenzylamine. $C_8H_{11}ON$. MW, 137. Liq. B.p. 224°/724 mm. Sol. H_2O, EtOH, Et_2O, C_6H_6. *B,HCl*: prisms from EtOH. M.p. 150°. *B_2,H_2PtCl_6*: yellow plates + $2H_2O$ from H_2O. M.p. 187°.

Et ether: $C_9H_{13}ON$. MW, 151. Oil. *B_2,H_2PtCl_6*: yellow cryst. M.p. 182°.

N-Acetyl: o-hydroxybenzylacetamide. Plates from $CHCl_3$–pet. ether. M.p. 140°.

Diacetyl deriv.: plates from $CHCl_3$–pet. ether. M.p. 102–3°.

Benzoyl: o-hydroxybenzylbenzamide. Plates from $CHCl_3$–pet. ether. M.p. 142° (softens at 140°).

Dibenzoyl deriv.: needles from EtOH–pet. ether. M.p. 142–3° (softens at 137°).

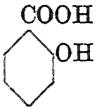
B,HCl: needles from EtOH. Very sol. H_2O.

B,HI: m.p. 184°.

B_2,H_2PtCl_6: yellow needles + $2H_2O$ from H_2O. M.p. 197° decomp.

Raiford, Clark, *J. Am. Chem. Soc.*, 1923, **45**, 1740.
Löw, *Monatsh.*, 1891, **12**, 397.
Tiemann, *Ber.*, 1890, **23**, 3017.
Goldschmidt, Ernst, *Ber.*, 1890, **23**, 2744.

Salicylic Acid (o-*Hydroxybenzoic acid*)



$C_7H_6O_3$ MW, 138

Occurs in form of esters in many essential oils and plant products, *e.g.* oil of wintergreen. Needles from H_2O. M.p. 159°. B.p. 211°/20 mm. Sol. EtOH, Et_2O, Me_2CO. Mod. sol. H_2O, C_6H_6. Volatile in steam. Sublimes in vacuo. Heat of comb. C_p 728·2 Cal. $k = 1{\cdot}06 \times 10^{-3}$ at 25°. Above 200° ⟶ phenol + CO_2 + a little salol. $FeCl_3$ ⟶ violet col. in H_2O. Strong antiseptic.

Me ester: *see* Methyl salicylate.

Et ester: *see* Ethyl salicylate.

Propyl ester: $C_{10}H_{12}O_3$. MW, 180. B.p. 238–40°. D^{20} 1·021.

Isopropyl ester: b.p. 120–2°/18 mm. D^{25} 1·0101. n_D^{25} 1·5003.

Butyl ester: $C_{11}H_{14}O_3$. MW, 194. M.p. 5·9°. B.p. 259–60°.

Isoamyl ester: *see* Isoamyl salicylate.

Allyl ester: $C_{10}H_{10}O_3$. MW, 178. B.p. 247–50°, 105°/5 mm. D^{15} 1·100.

Benzyl ester: $C_{14}H_{12}O_3$. MW, 228. B.p. 208°/26 mm., 186°/10 mm., 170–5°/7 mm. D_4^{20} 1·1799. n_D^{20} 1·5805.

2 : 4-*Dinitrobenzyl ester*: m.p. 168°.

Phenyl ester: *see* Salol.

o-*Tolyl ester*: $C_{14}H_{12}O_3$. MW, 228. M.p. 35°.

m-*Tolyl ester*: m.p. 74°.

p-*Tolyl ester*: m.p. 39°.

Salicyl ester: *see* Diplosal.

Phenacyl ester: m.p. 110°.

1-*Naphthyl ester*: *see under* 1-Naphthol.

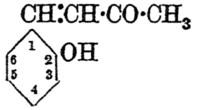
2-*Naphthyl ester*: *see under* 2-Naphthol.

Glycerol ester: trisalicylin. Needles. M.p. 79°.

Furfuryl ester: m.p. 131–2°.
Chloride: *o*-hydroxybenzoyl chloride. $C_7H_5O_2Cl$. MW, 156·5. Needles. M.p. 19–19·5°. B.p. 92°/15 mm.
Amide: $C_7H_7O_2N$. MW, 137. Yellow cryst. M.p. 133°. O-*Benzoyl*: m.p. 144°. N-*Benzoyl*: m.p. 208°.
Nitrile: *o*-cyanophenol. C_7H_5ON. MW, 119. Prisms from C_6H_6–pet. ether. M.p. 98°. $D_4^{99\cdot6}$ 1·1052. $n_\alpha^{99\cdot6}$ 1·53716. *Benzoyl*: needles from pet. ether. M.p. 106°.
Anilide: prisms from H_2O. M.p. 135°.
Piperidide: plates from EtOH. M.p. 142°.
Me ether: *see* *o*-Methoxybenzoic Acid.
Et ether: *see* *o*-Ethoxybenzoic Acid.
Propyl ether: $C_{10}H_{12}O_3$. MW, 180. Plates. M.p. 30°.
Benzyl ether: $C_{14}H_{12}O_3$. MW, 228. M.p. 76–7°.
Phenyl ether: *see* *o*-Phenoxybenzoic Acid.
Acetyl: *see* Acetylsalicylic Acid.
Benzoyl: needles. M.p. 132°.
p-*Nitrobenzoyl*: yellow cryst. from MeOH. M.p. 205°.

Schott, *Chem. Zentr.*, 1912, I, 754.
Ma, Hoo, Sah, *Chem. Abstracts*, 1934, **28**, 133.
Rosenmund, Harms, *Ber.*, 1920, **53**, 2230.
Pomilio, B.P. 103,739, (*Chem. Abstracts*, 1917, **11**, 1794).

Salicylideneacetone (o-*Hydroxybenzylideneacetone, methyl 2-hydroxystyryl ketone*)



$C_{10}H_{10}O_2$ MW, 162

Needles from ligroin. M.p. 140°. Sol. Et_2O. Spar. sol. cold H_2O.
Me ether: *o*-methoxybenzylideneacetone. $C_{11}H_{12}O_2$. MW, 176. Prisms. M.p. 48–50°. B.p. 180–2°/20 mm. $D_4^{61\cdot4}$ 1·0538. $n_D^{61\cdot4}$ 1·586. Sol. usual org. solvents.
Oxime: cryst. from C_6H_6–ligroin. M.p. 84–5°. Sol. Et_2O. Spar. sol. EtOH, C_6H_6, ligroin.
Semicarbazone: needles from EtOH. M.p. 206–7° decomp.

Tiemann, Kees, *Ber.*, 1885, **18**, 1966.
Harries, *Ber.*, 1891, **24**, 3180.
Auwers, *Ann.*, 1917, **413**, 279.

ω-Salicylideneacetophenone.
See 2-Hydroxychalkone.

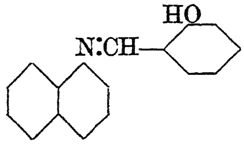
Salicylideneaniline (o-*Hydroxybenzylideneaniline, salicylaldehyde anil*)

$C_{13}H_{11}ON$ MW, 197

Yellow plates from MeOH. M.p. 51°. Sol. EtOH. Insol. H_2O.
B,HCl: yellow needles. M.p. 91°.
Me ether: *o*-methoxybenzylideneaniline. $C_{14}H_{13}ON$. MW, 211. Needles from EtOH. M.p. 44°. B.p. 330–4°, 235–6°/30 mm.
Picrate: yellow needles. M.p. 153–4°.

Schischkow, *Ann.*, 1857, **104**, 373.
Schiff, *Ann.*, 1869, **150**, 194.
Noelting, *Ann. chim.*, 1910, **19**, 540.

Salicylidene-1-naphthylamine (o-*Hydroxybenzylidene-1-naphthylamine*)

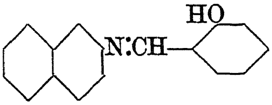


$C_{17}H_{13}ON$ MW, 247

Needles from C_6H_6. M.p. 53° (45·5°). Sol. EtOH, Et_2O, C_6H_6.
B,HCl: m.p. 210° decomp.

Pope, Fleming, *J. Chem. Soc.*, 1908, **93**, 1916.
Senier, Shepheard, *J. Chem. Soc.*, 1909, **95**, 443.

Salicylidene-2-naphthylamine (o-*Hydroxybenzylidene-2-naphthylamine*)



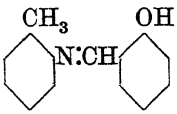
$C_{17}H_{13}ON$ MW, 247

Needles and prisms from EtOH. M.p. 126°. Sol. EtOH, C_6H_6, $CHCl_3$, ligroin. Spar. sol. Et_2O.

Senier, Shepheard, *J. Chem. Soc.*, 1909, **95**, 1950.
Emmerich, *Ann.*, 1887, **241**, 351.

1-Salicylidenepropionic Acid.
See α-Methyl-*o*-coumaric Acid *and* α-Methyl-*o*-coumarinic Acid.

Salicylidene-*o*-toluidine (o-*Hydroxybenzylidene*-o-*toluidine*)



$C_{14}H_{13}ON$ MW, 211

Needles from pet. ether. M.p. 47–8°.

Steinbrenck, *Ber.*, 1901, **34**, 833.

Salicylidene-*m*-toluidine (o-*Hydroxybenzylidene*-m-*toluidine*).
Yellowish prisms from MeOH. M.p. 39·5°.
Picrate: yellow needles from EtOH. M.p. 179°.

Senier, Shepheard, *J. Chem. Soc.*, 1909, **95**, 1945.

Salicylidene-*p*-toluidine (*o-Hydroxybenzylidene-p-toluidine*).

Cryst. from EtOH. M.p. 100°. Sol. EtOH, Et_2O.

Jaillard, *Compt. rend.*, 1865, **60**, 1098.

***o*-Salicyloylbenzoic Acid.**

See 2′-Hydroxybenzophenone-2-carboxylic Acid.

Salicyloylglycine.

See 2-Hydroxyhippuric Acid.

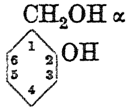
Salicyloylsalicylic Acid.

See Diplosal.

β-Salicyloylstyrene.

See 2′-Hydroxychalkone.

Saligenin (*Salicyl alcohol, o-hydroxybenzyl alcohol, ω-hydroxy-o-cresol*)



$C_7H_8O_2$ MW, 124

Needles or plates from H_2O or Et_2O. M.p. 87°. D^{25} 1·1613. Sol. EtOH, Et_2O. Mod. sol. H_2O, C_6H_6. Sublimes in plates. H_2SO_4 ⟶ red col. $FeCl_3$ ⟶ blue col. Antiseptic.

α-Me ether: $C_8H_{10}O_2$. MW, 138. B.p. 128–30°/40 mm.

2-Me ether: *o*-methoxybenzyl alcohol. B.p. 248–50°, 119°/8 mm. D^{17} 1·128. n_D^{17} 1·549. *Acetyl*: b.p. 130°/12 mm. D^{17} 1·117. n_D^{17} 1·515. *Benzoyl*: m.p. 59°.

Methylene ether: *see* 1 : 3-Benzdioxan.

Di-Me ether: $C_9H_{12}O_2$. MW, 152. B.p. 229–30°.

α-Et ether: $C_9H_{12}O_2$. MW, 152. B.p. 111–13°/20 mm.

2-Et ether: b.p. 265°.

2-Allyl ether: $C_{10}H_{12}O_2$. MW, 164. B.p. 133–50°/9 mm.

Diacetyl: b.p. 162–4°/11 mm., 103–4°/1 mm.

2-Benzoyl: needles from C_6H_6–pet. ether. M.p. 66°.

Dibenzoyl: fine needles from MeOH.Aq. M.p. 85°.

Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1680.
Rutovskii, Karolev, *Chem. Abstracts*, 1930, **24**, 24.
Manasse, *Ber.*, 1894, **27**, 2411.
Vavon, *Compt. rend.*, 1912, **154**, 359.
Paal, Senninger, *Ber.*, 1894, **27**, 1084.

Salinigrin.

See Piceoside.

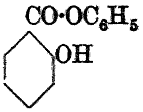
Salipurol.

See Naringenin.

Salipyrine.

See under Antipyrine.

Salol (*Phenyl salicylate*)



$C_{13}H_{10}O_3$ MW, 214

Plates from MeOH. M.p. 43° (41·7°). B.p. 173°/12 mm. D_4^{50} 1·1553. Sol. MeOH, EtOH, Et_2O. Insol. H_2O. Antiseptic. Phthalic anhydride + H_2SO_4 ⟶ phenolphthalein.

Me ether: $C_{14}H_{12}O_3$. MW, 228. Prisms from EtOH. M.p. 59°.

Phenyl ether: *see under* *o*-Phenoxybenzoic Acid.

p-Nitrobenzyl ether: cryst. from EtOH.Aq. M.p. 87°.

Chloroformyl: m.p. 90–1°.

Acetyl: cryst. from EtOH. M.p. 98°. B.p. 197–8°/11 mm.

Benzoyl: cryst. from EtOH. M.p. 80·5–81°.

Nicotinyl ester: colourless cryst. from Et_2O or EtOH. M.p. 91·5°.

Phosphate: $(HO)_2PO{\cdot}O{\cdot}C_6H_4{\cdot}CO{\cdot}OC_6H_5$. Cryst. M.p. 88°.

Phosphate, hydrate: $(HO)_4P{\cdot}O{\cdot}C_6H_4{\cdot}CO{\cdot}OC_6H_5$. Cryst. from C_6H_6. M.p. 62°. Very sol. H_2O. Sol. $CHCl_3$, AcOH, hot C_6H_6.

Phosphate diethyl ester: $(C_2H_5O)_2OP{\cdot}O{\cdot}C_6H_4{\cdot}CO{\cdot}OC_6H_5$. Oil. B.p. 105–13°/13 mm.

Phosphate diphenyl ester: $(C_6H_5O)_2OP{\cdot}O{\cdot}C_6H_4{\cdot}CO{\cdot}OC_6H_5$. Cryst. from EtOH. M.p. 76–7°.

Seifert, *J. prakt. Chem.*, 1885, **31**, 472.
Graebe, Eichengrün, *Ann.*, 1892, **269**, 324.
Chem. Fabr. v. Heyden, D.R.P., 38,973.
Bayer, D.R.P., 206,055, (*Chem. Zentr.*, 1909, I, 703).

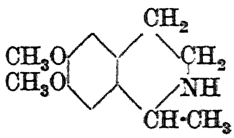
Salophene.

See *p*-Acetylaminophenyl salicylate.

Saloquinine.

See under Quinine.

Salsolidine (*O-Methylsalsoline, 6 : 7-dimethoxy-1-methyl-1 : 2 : 3 : 4-tetrahydroisoquinoline*)



$C_{12}H_{17}O_2N$ MW, 207

l-.

Alkaloid from *Salsola richteri*. Plates from H_2O. M.p. 47·5–48·5° (60–2°, anhyd. 71–3°). B.p. about 120°/0·01 mm. $[\alpha]_D^{15}$ − 59·7° in EtOH.

B,HCl: plates from H_2O. M.p. 235–6°. $[\alpha]_D^{15}$ − 24·8° in H_2O.

N-*Benzoyl*: needles from Me_2CO. M.p. 124–5°.

Picrate: yellow cryst. from EtOH. M.p. 194–5°.

Picrolonate: cryst. from EtOH. M.p. 235·5–236° (220–1°).

d-.

M.p. 47·5–48·5°. $[\alpha]_D^{15}$ + 59·9° in EtOH.

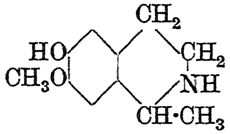
B,HCl: m.p. 235–6°. $[\alpha]_D^{15}$ + 25·3° in H_2O.

Picrate : m.p. 193–4°.
Picrolonate : m.p. 235·5–236°.

dl-.
M.p. 53–53·5°. B.p. about 140°/1 mm.
B,HCl : m.p. 196–7°.
N-*Benzoyl* : cryst. from EtOH.Aq. M.p. 127–8°.
Picrate : cryst. from MeOH. M.p. 201–201·5° decomp.
Picrolonate : cryst. from MeOH. M.p. 241° decomp.

Späth, Dengel, *Ber.*, 1938, **71**, 114.
Proskurnina, Orekhov, *Bull. soc. chim.*, 1937, **4**, 1265.

Salsoline (6-*Hydroxy*-7-*methoxy*-1-*methyl*-1 : 2 : 3 : 4-*tetrahydroisoquinoline*)

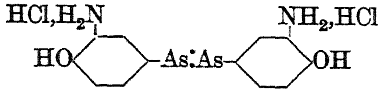


$C_{11}H_{15}O_2N$ MW, 193

Alkaloid from *Salsola richteri*. Micro-cryst. powder. M.p. 218–21°. Sol. EtOH, $CHCl_3$. Spar. sol. H_2O, C_6H_6. Insol. pet. ether. Sol. aq. alkalis.
B,HCl : needles + $1\frac{1}{2}H_2O$. M.p. 141–52°.
Benzoyl deriv. : cryst. from EtOH. M.p. 172–4°.
Dibenzoyl : cryst. from EtOH. M.p. 166–8°.
Me ether : *see* Salsolidine.

Orekhov, Proskurnina, *Ber.*, 1933, **66**, 841; 1934, **67**, 878.
Späth, Orekhov, Kuffner, *Ber.*, 1934, **67**, 1214.

Salvarsan (*Arsphenamine*, "606," *dihydrochloride of* 3 : 3′-*diamino*-4 : 4′-*dihydroxyarsenobenzene*)



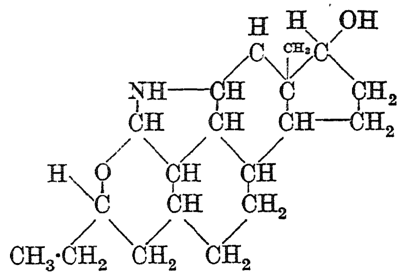
$C_{12}H_{14}O_2N_2Cl_2As_2$ MW, 439

Greyish powder. Decomp. (indefinite) at 180–95°. Sol. hot H_2O. Spar. sol. MeOH, EtOH. Insol. Et_2O, C_6H_6. Used in treatment of protozoal diseases.

Kirchoff, Korsina, Sirkin, D.R.P., 592,870, (*Chem. Zentr.*, 1934, I, 2791).
Christiansen, *J. Am. Chem. Soc.*, 1923, **45**, 1807.
Fargher, Pyman, *J. Chem. Soc.*, 1920, **117**, 376.

Salvianin chloride.
See Monardaein chloride.

Samandarin



$C_{19}H_{31}O_2N$ MW, 305

Secretion of *Salamander maculosa*, *Salamander atra*, etc. Colourless cryst. from EtOH.Aq., Me_2CO, and with 1MeOH from MeOH. M.p. 187–8°. Strongly basic. $[\alpha]_D^{17}$ + 43·7°. HCl ⟶ blue col.
B,HCl : m.p. 321–2°.
N-*Me* : $C_{20}H_{33}O_2N$. MW, 319. *B,HCl* : cryst. from EtOH.Aq. M.p. 300–2° decomp. *Methiodide* : cryst. from H_2O. M.p. 271–2°.
N-*Nitroso* : cryst. from EtOH.Aq. M.p. 164–5°.
Monoformyl deriv. : cryst. from MeOH.Aq. M.p. 148–50°.
Diformyl deriv. : needles from MeOH. M.p. 256–8°.
Diacetyl deriv. : needles from EtOH.Aq. M.p. 167–8°.

Schöpf, Braun, *Ann.*, 1934, **514**, 69.
Schöpf, Koch, *Ann.*, 1942, **62**, 552.

Sambunigrin (d-*Glucoside of* d-*mandelic acid nitrile*)

$C_6H_5{\cdot}CH{\cdot}O{\cdot}C_6H_{11}O_5$
CN

$C_{14}H_{17}O_6N$ MW, 295

Occurs in black Holunder. Colourless cryst. from C_6H_6–amyl alcohol. M.p. 151–152·5°. $[\alpha]_D^{18}$ − 75·1°. Emulsin ⟶ *d*-mandelonitrile + *d*-glucose.
Tetra-acetyl : needles from EtOH. M.p. 125–6°. $[\alpha]_D^{22}$ − 52·5°.

Fischer, Bergmann, *Ber.*, 1917, **50**, 1063.

Sambunigrinic Acid (β-d-*Glucoside of* d-*mandelic acid*)

$C_6H_5{\cdot}CH{\cdot}O{\cdot}C_6H_{11}O_5$
COOH

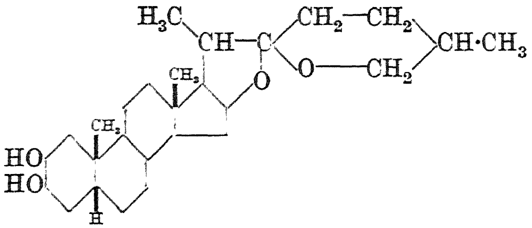
$C_{14}H_{18}O_8$ MW, 314

Needles from amyl alcohol, needles + 1EtOH from EtOH, m.p. 175–7°. Sol. H_2O, EtOH, Py. Spar. sol. Et_2O, Me_2O, AcOH, C_6H_6, $CHCl_3$. $[\alpha]_D^{18}$ + 51° in H_2O.
Quinine salt : prisms from H_2O. Sinters at 240°. M.p. 248° decomp. $[\alpha]_D^{20}$ − 70·66°.
Me ester : $C_{15}H_{20}O_8$. MW, 328. Needles from $CHCl_3$–CCl_4. M.p. 88–9°. Sol. H_2O. $[\alpha]_D^{19}$ + 41·2°.

Tetra-acetyl: needles from EtOH.Aq. M.p. 166°. $[\alpha]_D - 5{\cdot}34°$.

Fischer, *Z. physiol. Chem.*, 1919, **107**, 176; *Ber.*, 1919, **52**, 200.
Karrer, Nägeli, Weidmann, *Helv. Chim. Acta*, 1919, **2**, 259, 431.

Samogenin

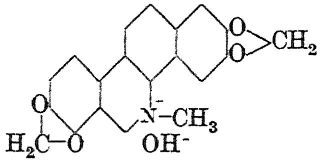


$C_{27}H_{44}O_4$ MW, 432

Present in *Samuela carnerosana*. M.p. 210–12°.
Diacetyl: m.p. 195–8°.

Marker *et al.*, *J. Am. Chem. Soc.*, 1943, **65**, 1199.

Sanguinarine (*ψ-Chelerythrine*)



Suggested structure

$C_{20}H_{15}O_5N$ MW, 349

Present in blood-root of *Sanguinaria canadensis*. Cryst. from Et_2O, m.p. 266° (242–3° slow heat.). Cryst. as alcoholate from alcoholic media, m.p. 195–7°. Sol. most org. solvents, sols. showing bluish-violet fluor.

Bruchhausen, Bersch, *Ber.*, 1930, **63**, 2520.
Späth, Kuffner, *Ber.*, 1931, **64**, 370, 1123, 2034.

Sanguisorbigenin

$C_{30}H_{46}O_3$ MW, 454

M.p. 275–6°. Heat ⟶ sanguisorbigenol, a triterpene alcohol.
Me ester: $C_{31}H_{48}O_3$. MW, 468. M.p. 207–9°. *Acetyl*: m.p. 243–5°.
Acetyl: m.p. 324°.

Matsukawa, *Chem. Abstracts*, 1935, **29**, 3346.

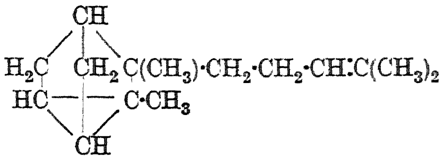
Santal

$C_{13}H_{10}O_4$ MW, 230

Plates or needles from EtOH. M.p. 222–3°. Sol. NaOH. $FeCl_3$ ⟶ reddish-violet col.
Di-Me ether: $C_{15}H_{14}O_4$. MW, 258. M.p. 141°.
Diacetyl: colourless cryst. from MeOH. M.p. 152°.

Raudnitz, Perlmann, *Ber.*, 1935, **68**, 1874.
O'Neill, Perkin, *J. Chem. Soc.*, 1918, **113**, 137.

α-Santalene

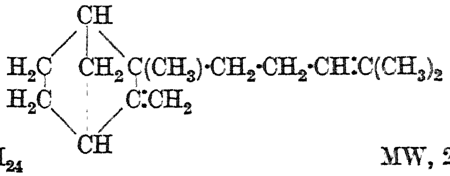


$C_{15}H_{24}$ MW, 204

Constituent of sandalwood oil. B.p. 252°/753 mm., 118–19°/9 mm. D^{20} 0·9118. n_D^{20} 1·4884. $[\alpha]_D - 15°$, $[\alpha]_{5780} + 2{\cdot}06°$. S ⟶ eudalene.
Dihydrochloride: b.p. 141–2°/0·5 mm. D^{20} 1·076. n_D^{20} 1·4976. $[\alpha]_D + 6°$.
Nitrosochloride: m.p. 112–17°.

Semmler, *Ber.*, 1910, **43**, 1898; 1907, **40**, 3322.
Schimmel, *Chem. Zentr.*, 1910, II, 1757.

β-Santalene



$C_{15}H_{24}$ MW, 204

Constituent of sandalwood oil. Oil with cedar-like odour. B.p. 125–7°/9 mm. D^{20} 0·8931. n_D^{20} 1·4959. $[\alpha]_D - 35°$.
Dihydrochloride: same as from α-santalene (above).
Nitrosochloride: two forms. (i) Plates. M.p. 152°. (ii) Needles. M.p. 106°.

Semmler, *Ber.*, 1910, **43**, 1898; 1907, **40**, 3322.
Schimmel, *Chem. Zentr.*, 1910, II, 1757.

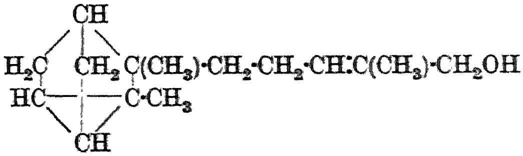
γ-Santalene

$C_{15}H_{24}$ MW, 204

Liq. B.p. 118°/10 mm. D^{20} 0·9355. n_D 1·5042.

Semmler, Bode, *Ber.*, 1907, **40**, 1130.

α-Santalol



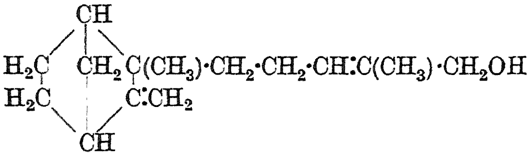
$C_{15}H_{24}O$ MW, 220

Constituent of sandalwood oil. B.p. 166–7°/14 mm., 143°/5 mm., 106°/0·06 mm. D_{25}^{25} 0·9970. n_D^{25} 1·5017. α_{5460} + 11·1° (+ 10·3°).
Allophanate: needles from MeOH. M.p. 162–3°.

Strychnine salt of hydrogen phthalate: prisms from AcOEt or Me_2CO. M.p. 144–5°. $[\alpha]_{5461}$ − 5·62° in C_6H_6.

Bradfield, Penfold, Simonsen, *J. Chem. Soc.*, 1935, 309.
Ruzicka, Thomann, *Helv. Chim. Acta*, 1935, **18**, 357.

β-Santalol



$C_{15}H_{24}O$ MW, 220

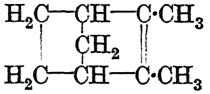
Constituent of sandalwood oil. B.p. 177–8°/17 mm., 112°/0·06 mm. D_{25}^{25} 0·9729. n_D^{25} 1·5117. α_{5461} − 87·1°.

Allophanate: needles from MeOH. M.p. 159–60°.

Strychnine salt of hydrogen phthalate: prisms from AcOEt. M.p. 134–5°. $[\alpha]_{5461}$ − 37·5° in C_6H_6.

Bradfield, Penfold, Simonsen, *J. Chem. Soc.*, 1935, 309.
Ruzicka, Thomann, *Helv. Chim. Acta*, 1935, **18**, 357.

Santene



C_9H_{14} MW, 122

Constituent of sandalwood oil and pine-needle oil. Liq. with odour resembling camphene. B.p. 140–1°, 35°/15 mm. D_4^{17} 0·8698. n_D^{17} 1·4688.

Nitrosite: three forms. (i) Blue. M.p. 122–4°. (ii) Colourless. M.p. 104°. (iii) Green. M.p. 127–8° decomp.

Nitrosate: m.p. 216° decomp.

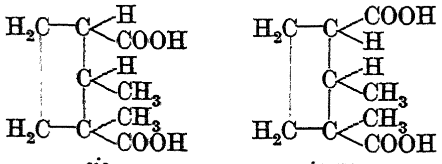
Nitrosochloride: blue. M.p. 109–10°. A white cryst. polymer is also described.

Diels, Alder, *Ann.*, 1931, **486**, 209.
Ruzicka, Liebl, *Helv. Chim. Acta*, 1923, **6**, 271.
Komppa, Hintikka, *Bull. soc. chim.*, 1917, **21**, 14.

Santene Hydrate.

See β-Santenol.

Santenic Acid (1 : 2-*Dimethylcyclopentane*-1 : 3-*dicarboxylic acid*)



cis-. *trans*-.

Probable configurations

$C_9H_{14}O_4$ MW, 186

Cis:

Acid from natural sources, by oxidation of santenol or santenone. Plates from H_2O. M.p. 170–1°. $[\alpha]_D^{33}$ + 38·31° in EtOH. AcOH–HCl ⟶ *trans*-form.

Di-Me ester: $C_{11}H_{18}O_4$. MW, 214. B.p. 120–3°/9 mm. D^{20} 1·078. n_D^{20} 1·4645.

Di-Et ester: $C_{13}H_{22}O_4$. MW, 242. B.p. 143–4°/14 mm. D_4^{20} 1·0268. n_D^{20} 1·44854.

Anhydride: $C_9H_{12}O_3$. MW, 168. Cryst. M.p. 129–30°.

Anil: $C_{15}H_{17}O_2N$. MW, 243. Cryst. from AcOH.Aq. M.p. 117–18°.

Monoanilide: cryst. from AcOH.Aq. M.p. 205–6°.

Trans:

Cryst. from H_2O. M.p. 166–7°.

Dianilide: needles from EtOH–C_6H_6 or EtOH.Aq. M.p. 221°.

Komppa, *Chem. Zentr.*, 1936, I, 3838; *Ber.*, 1932, **65**, 1708.
Enkvist, *J. prakt. Chem.*, 1933, **137**, 261.
Komppa, Rohrmann, *Ber.*, 1934, **67**, 828.
Komppa, Hintikka, *Bull. soc. chim.*, 1917, **21**, 17.
Ishidate, Sano, *Ber.*, 1941, **74**, 1189.

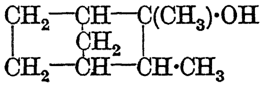
Santenol.

See under Norborneol.

α-Santenol.

See under Norborneol.

β-Santenol (*Santene hydrate*)

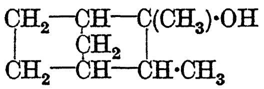


$C_9H_{16}O$ MW, 140

Needles from EtOH. M.p. 101–2°. B.p. 192°. Stereoisomeric with γ-santenol.

Diels, Alder, *Ann.*, 1931, **486**, 205.

γ-Santenol



$C_9H_{16}O$ MW, 140

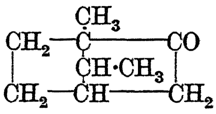
M.p. 63–5°. B.p. 82–4°/15 mm. Stereoisomeric with β-santenol.

Diels, Alder, *Ann.*, 1931, **486**, 209.

Santenone.

See under Norcamphor.

α-Santenone



$C_9H_{14}O$ MW, 138

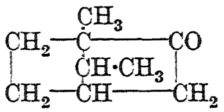
M.p. 55°. B.p. 191°.

Oxime: m.p. 74°.

Semicarbazone: m.p. 236°.

Aschan, *Chem. Abstracts*, 1934, **28**, 2344.
Asahina, Ishidate, *Ber.*, 1935, **68**, 950.

β-Santenone



$C_9H_{14}O$ MW, 138

M.p. 49–50°. B.p. 190°.

Oxime: m.p. 51°.

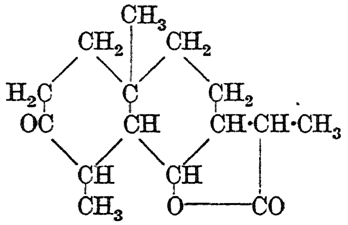
Semicarbazone: m.p. 236–7°.

Aschan, *Chem. Abstracts*, 1934, **28**, 2344.
Asahina, Ishidate, *Ber.*, 1935, **68**, 950.
Mukherji, *Chem. Abstracts*, 1949, **43**, 3796.

Santenone Alcohol.
See under Norborneol.

Santenylamine.
See under Norbornylamine.

Santonan (*Tetrahydrosantonin*)



$C_{15}H_{22}O_3$ MW, 250

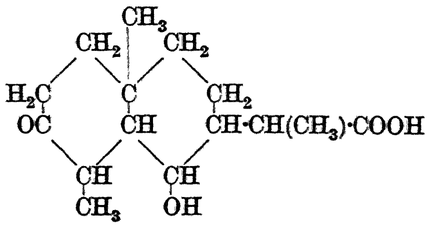
Two forms.

α-.
Cryst. from EtOH. M.p. 158°. $[\alpha]_D^{18}$ + 17·8° in MeOH.
Oxime: cryst. from EtOH. M.p. 219–20°.
Semicarbazone: powder. M.p. 258° decomp.
Phenylhydrazone: cryst. from EtOH. M.p. 205° decomp.

β-.
Plates from Et_2O. M.p. 105°. $[\alpha]_D^{18}$ + 9·3° in MeOH. More sol. than α-form.
Oxime: m.p. 182°.
Semicarbazone: m.p. 250° decomp.

Clemo, Haworth, *J. Chem. Soc.*, 1930, 2580.
Wedekind, Goost, Jäckh, *Ber.*, 1930, **63**, 50.
Wienhaus, Oettingen, *Ann.*, 1913, **397**, 240.
Asahina, *Ber.*, 1913, **46**, 1776.

Santonanic Acid (*Tetrahydrosantoninic acid*)



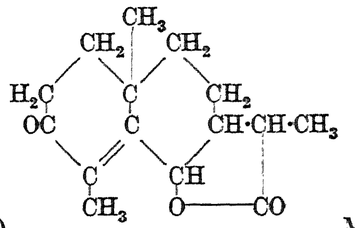
$C_{15}H_{24}O_4$ MW, 268

α-.
Cryst. + $1H_2O$. Sinters at 85°. M.p. 115° decomp. $[\alpha]_D^{18}$ + 20·0°. Heat above m.p. ⟶ α-lactone.

β-.
Cryst. from H_2O. M.p. about 192°. $[\alpha]_D^{18}$ + 2·2°. Heat above m.p. ⟶ β-lactone.
Oxime: m.p. 218–20°.

Wienhaus, Oettingen, *Ann.*, 1913, **397**, 238.

Santonene (*Dihydrosantonin*)



$C_{15}H_{20}O_3$ MW, 248

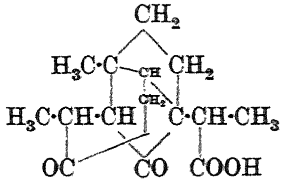
Two forms.

(i) Prisms or needles from EtOH.Aq. M.p. 105°. $[\alpha]_D^{18}$ + 75·3° in EtOH.
Oxime: prisms from EtOH. M.p. 235° decomp.
Semicarbazone: prisms from EtOH. M.p. 245° decomp.

(ii) Needles from EtOH. M.p. 181–2°. $[\alpha]_D^{25}$ — 280·9°. Insol. Na_2CO_3.
Oxime: needles. M.p. 196°. $[\alpha]_D^{25}$ — 239° in EtOH.

Wedekind, Goost, Jäckh, *Ber.*, 1930, **63**, 53.
Medvedev, Alekseeva, *Chem. Abstracts*, 1928, **22**, 1979.

Santonic Acid



$C_{15}H_{20}O_4$ MW, 264

Cryst. from H_2O or EtOH. M.p. 170–2°. B.p. 285°/15 mm. $[\alpha]_D^{20}$ — 74·0° in $CHCl_3$. Sol. 190 parts H_2O at 17°. Dist. ⟶ meta-santonic acid. H_2SO_4 ⟶ santonin.

Me ester: $C_{16}H_{22}O_4$. MW, 278. Prisms from MeOH, needles from EtOH.Aq. M.p. 86°. $[\alpha]_D^{25}$ — 52·3° in $CHCl_3$. *Oxime*: prisms. M.p. 158–9°.

Et ester: $C_{17}H_{24}O_4$. MW, 292. Prisms from Et_2O. M.p. 94–5°. $[\alpha]_D^{25}$ — 45·4° in $CHCl_3$. *Oxime*: plates from EtOH. M.p. 126°. *Monophenylhydrazone*: yellow plates. M.p. 115°.

Propyl ester: $C_{18}H_{26}O_4$. MW, 306. Syrup. B.p. 220°/3 mm.

Isobutyl ester: $C_{19}H_{28}O_4$. MW, 320. Needles from Et_2O. M.p. 67°.

Chloride: $C_{15}H_{19}O_3Cl$. MW, 282·5. Prisms from EtOH. M.p. 170–1°.

Bromide: $C_{15}H_{19}O_3Br$. MW, 327. M.p. 145·5°.

Iodide: $C_{15}H_{19}O_3I$. MW, 374. Needles from Et_2O. M.p. 136°.

Oxime: cryst. from EtOH. M.p. 186°.

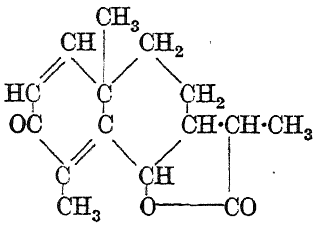
Dioxime: m.p. 120–5° decomp.

Monosemicarbazone: m.p. 183–5° decomp.

Monophenylhydrazone: yellow needles from EtOH. M.p. 174°.

Abkin, Medvedev, *Chem. Abstracts*, 1935, **29**, 3682.
Harries, Stähler, *Ber.*, 1904, **37**, 259.
Francesconi, *Gazz. chim. ital.*, 1899, **29**, 224.
Woodward, Brutschy, Baer, *J. Am. Chem. Soc.*, 1948, **70**, 4216.

Santonin



$C_{15}H_{18}O_3$ MW, 246

Widely occurring in plants, especially in *Artemisia*. Colourless cryst. from H_2O, EtOH or Et_2O. M.p. 174–6°. $[\alpha]_D^{18}$ − 173·0° in EtOH. Turns yellow in sunlight. Isomerises in sol. in daylight. $H_2SO_4 \longrightarrow$ blue col. EtOH-KOH $\longrightarrow$ red col. Zn + AcOH $\longrightarrow$ santonone. Zn dust dist. $\longrightarrow$ 1 : 4-dimethylnaphthalene and 1 : 4-dimethyl-2-naphthol. Alkalis $\longrightarrow$ salts of santonic acid. Sol. has bitter taste. Used in treatment of nervous complaints.

Oxime: needles + $1H_2O$ from EtOH.Aq. M.p. 218°.

Semicarbazone: cryst. from AcOEt. M.p. 232° decomp.

Phenylhydrazone: yellow needles. M.p. 230–1° decomp.

Clemo, Haworth, *J. Chem. Soc.*, 1930, 2579.
Ruzicka, Eichenberger, *Helv. Chim. Acta*, 1930, **13**, 1117.
Wedekind, Tettweiler, *Ber.*, 1931, **64**, 1796.
Massagetov, *Chem. Abstracts*, 1932, **26**, 4413.
Cornforth, Cornforth, Dewar, *Nature*, 1944, **153**, 317.

β-Santonin

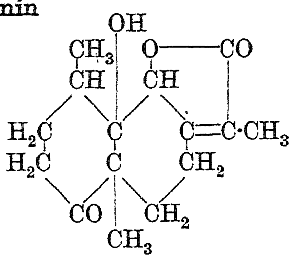
$C_{15}H_{18}O_3$ MW, 246

Stereoisomeric with santonin. Constituent of certain varieties of *Artemisia*. Colourless prisms from EtOH. M.p. 216–18°. $[\alpha]_D^{19}$ − 137·2° in $CHCl_3$.

Oxime: prisms from EtOH. M.p. 224°.

Clemo, *J. Chem. Soc.*, 1934, 1343.

ψ-Santonin



Suggested structure

$C_{15}H_{20}O_4$ MW, 264

Rhombic cryst. M.p. 183°. $[\alpha]_D^{20}$ − 169° in $CHCl_3$.

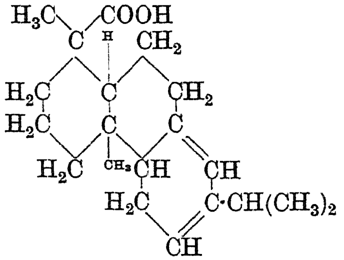
Acetyl: m.p. 187°. $[\alpha]_D^{20}$ − 69°.

Oxime: m.p. 203–4°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 257°.

Clemo, Cocker, *J. Chem. Soc.*, 1946, 30.
Cocker, Cross, Lipman, *J. Chem. Soc.*, 1949, 959.
Cocker, Cross, Hayes, *Chemistry and Industry*, 1952, 314.

***l*-Sapietic Acid** (*Levopimaric acid*)



$C_{20}H_{30}O_2$ MW, 302

Occurs in *Pinus palustris*, etc. Prisms from EtOH. M.p. 152° (148–51°). $[\alpha]_D^{20}$ − 282° in EtOH. Isomerised by hot AcOH to abietic acid. Absorption maximum at 272·5 mμ.

Me ester: $C_{21}H_{32}O_2$. MW, 316. M.p. 63–4°. $[\alpha]_D$ − 268° in Et_2O.

Et ester: $C_{22}H_{34}O_2$. MW, 330. B.p. 175–7°/0·5 mm. D_4^{23} 1·0124. n_D^{23} 1·5153. $[\alpha]_D$ − 170·9° in EtOH.

Hasselstrom, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 2118.
Palkin, Morris, *J. Am. Chem. Soc.*, 1933, **55**, 3677.
Ruzicka, Balos, Vilim, *Helv. Chim. Acta*, 1924, **7**, 458.
Harris, Sanderson, *J. Am. Chem. Soc.*, 1948, **70**, 334.
Simonsen, Barton, *The Terpenes*, Vol. III, 428, (Cambridge University Press, 1952).

Saponarin (*Vitexin glucoside*)

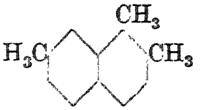
$C_{21}H_{24}O_{12}$ MW, 468

Occurs in *Saponaria officinalis*, Linn., and other plants. Needles + $2H_2O$ from Py.Aq. M.p. 231–2° decomp. (slow heat), 236° (rapid heat). $[\alpha]_D$ − 7·9° in H_2O. Sol. alkalis with yellow col. Conc. H_2SO_4 ⟶ blue fluor. I ⟶ blue col. which disappears on dilution or addition of EtOH or on warming; reappears on cooling.

Nona-acetyl deriv.: m.p. 183–5°.

Molisch, *Chem. Abstracts*, 1912, **6**, 766.
Barger, Field, *J. Chem. Soc.*, 1912, **101**, 1396.

Sapotalin (1 : 2 : 7-*Trimethylnaphthalene*)



$C_{13}H_{14}$ MW, 170

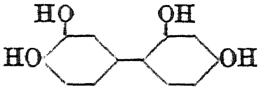
B.p. 148°/16 mm. D_4^{15} 1·008. n_D^{15} 1·6093.

Picrate: m.p. 129·5–130°.

Styphnate: m.p. 156°.

Spring, Vickerstaff, *J. Chem. Soc.*, 1937, 252.

Sappanin (2 : 4 : 3′ : 4′-*Tetrahydroxydiphenyl*)



$C_{12}H_{10}O_4$ MW, 218

Occurs in *Cæsalpinia Sappan*, Linn. Leaflets + $2H_2O$ from H_2O. M.p. anhyd. 210–11°. B.p. 230–40°/0·01 mm. Sol. EtOH, Et_2O, hot H_2O. Insol. $CHCl_3$, CS_2, C_6H_6. $FeCl_3$ ⟶ red. col. Reduces Fehling's and Tollen's on warming.

Tetra-Me ether: $C_{16}H_{18}O_4$. MW, 274. Cryst. from MeOH.Aq. M.p. 74–5°.

Späth, Gibian, *Monatsh.*, 1930, **55**, 342.

Sarcine.

See Hypoxanthine.

Sarcolactic Acid.

See under Lactic Acid.

Sarcosine (*Methylaminoacetic acid*, N-*methylglycine*, N-*methylglycocoll*)

$CH_3{\cdot}NH{\cdot}CH_2{\cdot}COOH$

$C_3H_7O_2N$ MW, 89

Cryst. from EtOH. M.p. 212–13° decomp. Heat of comb. C_v 401·2 Cal.

B,HCl: m.p. 168–70°.

B,HBr: m.p. 186–7°.

B,HI: m.p. 152°.

B_2,H_2PtCl_6: m.p. 193–4° decomp.

Et ester: $C_5H_{11}O_2N$. MW, 117. B.p. 43°/10 mm. $D^{19·5}$ 0·971. *Picrate*: m.p. 149·5°.

Nitrile: $C_3H_6N_2$. MW, 70. Viscous liq. *B_2,H_2SO_4*: does not melt below 210°.

N-Benzoyl: *N* methylhippuric acid. M.p. 103·5–104° decomp.

N-Phenyl: *see* N-Phenylsarcosine.

Baumann, *J. Biol. Chem.*, 1915, **21**, 563.
Cocker, Lapworth, *J. Chem. Soc.*, 1931, 1894.
Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, **36**, 372.
Schütte, *Z. physiol. Chem.*, 1943, **279**, 61.

Sarmentocymarin

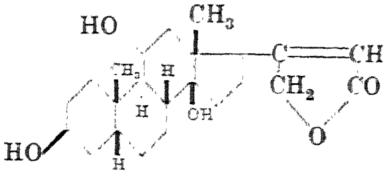
$C_{30}H_{46}O_8$ MW, 534

Occurs in *Strophanthus* seeds as glycoside. M.p. 130°. $[\alpha]_D^{20}$ − 12·5°. Hyd. ⟶ sarmentogenin and sarmentose.

Dibenzoyl deriv.: m.p. 281°. $[\alpha]_D^{20}$ − 14°.

Jacobs, Heidelberger, *J. Biol. Chem.*, 1929, **85**, 765.
Katz, *Helv. Chim. Acta*, 1948, **31**, 993.

Sarmentogenin



$C_{23}H_{34}O_5$ MW, 390

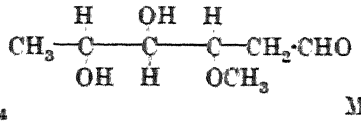
Isolated from seeds of *Strophanthus sarmentosus*. Needles from EtOH. M.p. 270°. $[\alpha]_D$ + 21·3° in EtOH.

3 : 11-*Diacetyl deriv.*: m.p. 120–60°.

Dibenzoyl deriv.: hexagonal prisms from Me_2CO.Aq. M.p. 281°. $[\alpha]_D^{20}$ + 14° in Me_2CO.

Jacobs, Heidelberger, *J. Biol. Chem.*, 1929, **81**, 765.
Mason, Hoehn, *J. Am. Chem. Soc.*, 1938, **60**, 2824.
Tschesche, Bohle, *Ber.*, 1936, **69**, 2497.
Katz, *Helv. Chim. Acta*, 1948, **31**, 993.
Callow, Meikle, Taylor, *Chemistry and Industry*, 1951, 336.

Sarmentose (3 : 4-*Dihydroxy-2-methoxycaproic aldehyde*)



$C_7H_{14}O_4$ MW, 162

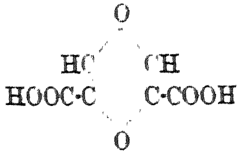
Occurs in cardiac glycosides. M.p. 78–9°. $[\alpha]_D$ + 16° in H_2O.

α-Methylsarmentoside: m.p. 33–6°. $[\alpha]_D^{20}$ + 156° in Me_2CO.

β-Methylsarmentoside: m.p. 35–45°. $[\alpha]_D^{20}$ − 39° in Me_2CO.

Hauenstein, Reichstein, *Helv. Chim. Acta*, 1950, **33**, 446.

Sarsapic Acid (*Sarsapinic acid*)



Probable structure

$C_6H_4O_6$ MW, 172

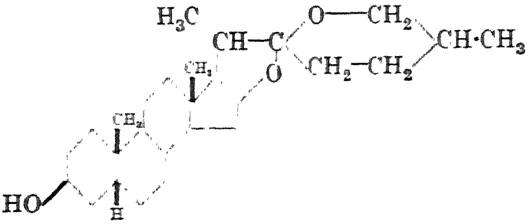
Occurs in sarsaparilla root. Needles from hot H_2O. M.p. 305°. Sol. EtOH. Spar. sol. Et_2O.

Di-Me ester: $C_8H_8O_6$. MW, 200. Leaflets from EtOH. M.p. 121°.

Power, Salway, *J. Chem. Soc.*, 1914, **105**, 205.

Sarsapinic Acid.
See Sarsapic Acid.

Sarsasapogenin (*Parigenin*)



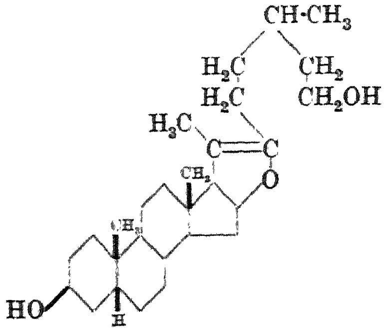
$C_{27}H_{44}O_3$ MW, 416

Occurs in Vera-Cruz sarsaparilla root. Cryst. from Me_2CO. M.p. 200–201·5°. $[\alpha]_D^{25}$ − 75° in $CHCl_3$.

Acetyl: m.p. 144–5°. $[\alpha]_D^{25}$ − 70·2° in $CHCl_3$.
Benzoyl: m.p. 170–1°.
o-*Bromobenzoyl*: m.p. 128–9°.
α-*Glucoside*: m.p. 245°. *Tetra-acetyl*: cryst. from EtOH. M.p. 227°.

Farmer, Kon, *J. Chem. Soc.*, 1937, 414.
Marker, *J. Am. Chem. Soc.*, 1941, **63**, 2274.
Fieser and Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 580–91.

ψ-Sarsasapogenin



$C_{27}H_{44}O_3$ MW, 416

Needles from AcOEt. M.p. 171–3°. Sol. Et_2O.

Di-p-nitrobenzoyl: pale yellow cryst. from Me_2CO. M.p. 156·5–159°.

Marker, Rohrmann, *J. Am. Chem. Soc.*, 1940, **62**, 518, 521.
Marker, Jones, Krueger, *ibid.*, 2532.
Marker *et al.*, *J. Am. Chem. Soc.*, 1942, **64**, 1655.

***epi*-Sarsasapogenin**

$C_{27}H_{44}O_3$ MW, 416

Prepared by red. with sodium-amyl alcohol of the ketone from sarsasapogenin. Needles from Me_2CO. M.p. 207°.

Acetyl: plates. M.p. 194°.

Marker, Rohrmann, *J. Am. Chem. Soc.*, 1939, **61**, 943.

Satisterol

"$C_{27}H_{46}O$" MW, 386

Phytosterol from rice embryo. M.p. 156°. $[\alpha]_D^{23}$ − 14·5°.

Acetyl: m.p. 111°. $[\alpha]_D^{23}$ − 9·7°.
Benzoyl: m.p. 129°. $[\alpha]_D^{20}$ − 14·5°.

Kimm, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1938, **34**, 637.
Barton, *J. Chem. Soc.*, 1946, 512, 517.

Sativic Acid (*Sativinic acid*, 8 : 9 : 11 : 12-*tetrahydroxystearic acid*)

$CH_3{\cdot}[CH_2]_4{\cdot}[CH(OH)]_2{\cdot}CH_2{\cdot}[CH(OH)]_2{\cdot}[CH_2]_7{\cdot}COOH$

$C_{18}H_{36}O_6$ MW, 348

Occurs in bark of various plants and in human fat. (α) M.p. 164°. (β) M.p. 174°. (γ) M.p. 144–5°. (δ) M.p. 135°.

Zellner, *Monatsh.*, 1925, **46**, 619.
Nicolet, Cox, *J. Am. Chem. Soc.*, 1922, **44**, 144.
Dreyfuss, F.P., 636,488, (*Chem. Abstracts*, 1929, **23**, 1140).
Wagner, *Biochem. Z.*, 1926, **174**, 412.
Reinger, *Chem. Abstracts*, 1922, **16**, 3467.
Birosel, *J. Am. Chem. Soc.*, 1937, **59**, 689.

Sativinic Acid.
See Sativic Acid.

Savinin

$C_{20}H_{14-16}O_6$ MW, 350–352

Constituent of *Juniperus sabina*. Cryst. from C_6H_6 or EtOH. M.p. 146·4–148·4°. $[\alpha]_D^{21}$ −87° (c = 0·95 in $CHCl_3$). Contains lactonic group. Insol. aq. alkalis.

Hartwell *et al.*, *J. Am. Chem. Soc.*, 1953, **75**, 235.

Saxatilic Acid.
See Salazinic Acid.

Scammonin.
See Jalapin.

Scatole.

See Skatole.

Schäffer Acid.

See 2-Naphthol-6-sulphonic Acid.

Schöllkopf Acid.

See 1-Naphthylamine-8-sulphonic Acid.

Scianthrene

$C_{18}H_{18}$ MW, 234

Cryst. from EtOH. M.p. 86–7°. Possibly 2-methyl-8-isopropylphenanthrene.

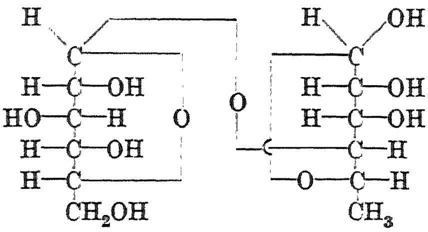
Picrate : m.p. 123–4°.

Styphnate : m.p. 138–9°.

sym.-*Trinitrobenzene add. comp.* : m.p. 146–7°.

Uota, *Chem. Abstracts*, 1937, **31**, 7416.
Orcutt, Bogert, *J. Am. Chem. Soc.*, 1941, **63**, 127.

Scillabiose (4-l-*Rhamnose* d-*glucoside*)



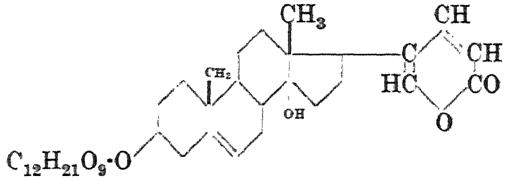
$C_{12}H_{22}O_{10}$ MW, 326

Obtained from Scillaren-A.

Phenylosazone : m.p. 164·5—165°.

Zemplén, *Chem. Abstracts*, 1939, **33**, 4202.

Scillaren-A

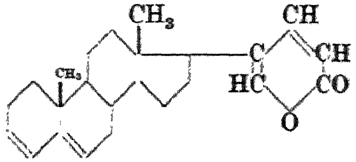


$C_{36}H_{52}O_{13}$ MW, 692

Occurs in squills. Plates + $1H_2O$ + 1MeOH from MeOH.Aq., m.p. 230–40°; needles from EtOH, m.p. 270°. $[\alpha]_D^{20}$ − 73·8° in EtOH. Hyd. ⟶ rhamnose and glucose.

Tschesche, Haupt, *Ber.*, 1937, **70**, 44.
Stoll, Hofmann, *Helv. Chim. Acta*, 1935, **18**, 82, 401.
Stoll *et al.*, *Helv. Chim. Acta*, 1933, **16**, 703 ; 1935, **18**, 649.

Scillaridin-A

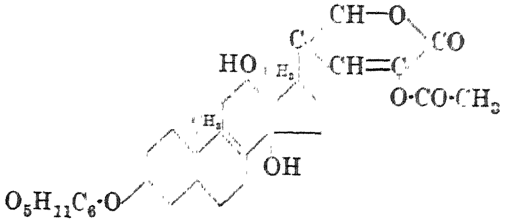


$C_{24}H_{30}O_3$ MW, 366

Prisms from EtOH. M.p. 245–50°. Spar. sol. EtOH, $CHCl_3$. Insol. H_2O, Et_2O. $[\alpha]_D^{20}$ − 62·6° in MeOH–$CHCl_3$.

Tschesche, Haupt, *Ber.*, 1937, **70**, 44.
Stoll *et al.*, *Helv. Chim. Acta*, 1933, **16**, 727 ; 1935, **18**, 647, 1247.

Scilliroside



Suggested structure

$C_{32}H_{44}O_{12}$ MW, 620

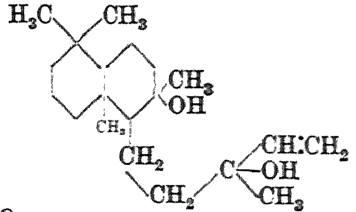
Occurs in red squill. M.p. 168—70°. $[\alpha]_D^{20}$ − 59·4°.

Tetra-acetyl : m.p. 199°. $[\alpha]_D^{20}$ − 48°.

Tetra-benzoyl : m.p. 168–70°.

Stoll, Renz, *Helv. Chim. Acta*, 1942, **25**, 43.
Stoll, Renz, Helfenstein, *Helv. Chim. Acta*, 1943, **26**, 648.
Fieser and Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 555–8.

Sclareol



$C_{20}H_{36}O_2$ MW, 308

Diterpene diol from leaves of *Salvia sclarea*, Linn. Needles from pet. ether. M.p. 105·5–106° corr. B.p. 182°/1 mm., 163–5°/0·25 mm. $[\alpha]_D^{18}$ − 6·25° in EtOH.

Ruzicka, Engel, Fischer, *Helv. Chim. Acta*, 1938, **21**, 364.
Ruzicka, Janot, *Helv. Chim. Acta*, 1931, **14**, 645.
Hosking, Brandt, *Ber.*, 1935, **68**, 37.
Janot, *Ann. chim.*, 1932, **17**, 5.
Ruzicka, Seidel, Engel, *Helv. Chim. Acta*, 1942, **25**, 621.

Scoparin

$C_{22}H_{22}O_{11}$ MW, 462

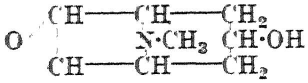
Occurs in *Cytisus scoparius*, Link. M.p. 230° decomp. Sol. hot H_2O, EtOH, Me_2CO. Insol. Et_2O, $CHCl_3$, CS_2. Reduces Fehling's.

Di-Me ether : $C_{24}H_{26}O_{11}$. MW, 490. Yellow cryst. M.p. 260–5° decomp.

Tri-Me ether : $C_{25}H_{28}O_{11}$. MW, 504. Yellow cryst. M.p. 220–38°.
Octa-Me ether : $C_{23}H_{32}O_{11}$. MW, 560. M.p. 120–30° ⟶ solid, remelting at 229–33°.

Hemmelmayr, Strehly, *Monatsh.*, 1926, **47**, 379.
Herzig, Tiring, *Monatsh.*, 1918, **39**, 253.
Mascré, Paris, *Compt. rend.*, 1937, **204**, 1270.

Scopine



$C_8H_{13}O_2N$ MW, 155

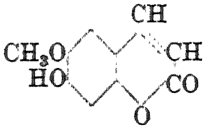
Needles from pet. ether. M.p. 76°.
B,HAuCl₄ : m.p. 216° decomp.
B₂,H₂PtCl₆ : m.p. 219° decomp.
Picrate : leaflets. M.p. 231°.
Tropic ester : *see* Hyoscine.

ψ-Scopine.
M.p. 125–6°.
Benzoyl : m.p. 142°. *B,HCl* : m.p. 216°. *Chloroaurate* : m.p. 220°. *Picrate* : m.p. 204°. *Phenylurethane* : m.p. 229°. *B,HAuCl₄* : m.p. 210°.

Willstätter, Berner, *Ber.*, 1923, **56**, 1079, 1081.
Polonovski, Polonovski, *Bull. soc. chim.*, 1928, **43**, 590, 594.

Scopolamine.
See Hyoscine.

Scopoletin (*Æsculetin* 6-*methyl ether, chrysatropic acid, gelseminic acid,* 7-*hydroxy*-6-*methoxycoumarin,* β-*methylæsculetin*)



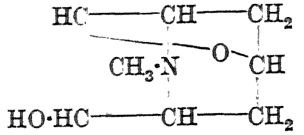
$C_{10}H_8O_4$ MW, 192

Occurs in root of *Gelsemium sempervirens*, Ait., *Atropa belladonna*, Linn., *Convolvulus scammonia*, Linn., *Ipomœa orizabensis*, Ledenois, *Prunus serotina*, Ehrh. and *Fabiana imbricata*, Ruiz & Pav. Needles or prisms. M.p. 204°. Sol. hot EtOH, hot AcOH. Mod. sol. $CHCl_3$. Spar. sol. H_2O. Insol. C_6H_6, CS_2. Blue fluor. in EtOH. Reduces Fehling's and Tollen's. $FeCl_3$ ⟶ green col.
Acetyl : m.p. 177°.
Me ether : 6 : 7-dimethoxycoumarin. $C_{11}H_{10}O_4$. MW, 206. M.p. 145°.
Glucoside : *see* Fabiatrin.

Seka, Kallir, *Ber.*, 1931, **64**, 909 (*Bibl.*).
Head, Robertson, *J. Chem. Soc.*, 1931, 1241 (*Bibl.*).

Scopoligenin.
See Norscopoline.

Scopoline (*Oscine*)

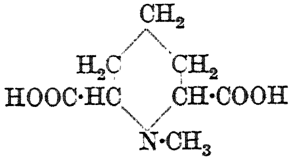


$C_8H_{13}O_2N$ MW, 155

Prisms from Et_2O. M.p. 108–9°. B.p. 248°.
B,HCl : m.p. 270°.
B,HAuCl₄ : m.p. 220° decomp.
B₂,H₂PtCl₆ : m.p. 203° decomp.
Picrate : m.p. 236°.

Willstätter, Berner, *Ber.*, 1923, **56**, 1079.
Polonovski, Polonovski, *Bull. soc. chim.*, 1928, **43**, 590.

Scopolinic Acid (*N-Methylhexahydrodipicolinic acid, N-methylpiperidine*-2 : 6-*dicarboxylic acid*)



$C_8H_{13}O_4N$ MW, 187

Plates + $1H_2O$. M.p. 225° decomp. (230° anhyd.). Sol. hot H_2O. Spar. sol. Et_2O.
B,HCl : m.p. 225–6° decomp.
Di-Me ester : $C_{10}H_{17}O_4N$. MW, 215. B.p. 140–1°/13 mm.

Schmidt, *Arch. Pharm.*, 1915, **253**, 606.
Hess, Wissing, *Ber.*, 1915, **48**, 1910.
Willstätter, Lessing, *Ber.*, 1902, **35**, 2072.
Hess, Suchier, *Ber.*, 1915, **48**, 2057.

Scopularic Acid.
See Stictic Acid.

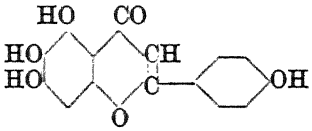
Scorodose

$C_{24}H_{42}O_{21}$ MW, 666

Tetrasaccharide from the bulbs of Allium. Probably contains four fructose residues.
Acetyl : m.p. 85–90°. $[\alpha]_D^{14}$ − 28·5°.
Benzoyl : m.p. 148°. $[\alpha]_D^{23}$ − 57·1° in $CHCl_3$.
Me ether : m.p. 45. $[\alpha]_D^{17}$ − 49·2° in EtOH.

Kihara, *Chem. Abstracts*, 1940, **34**, 385.

Scutellarein (5 : 6 : 7 : 4′-*Tetrahydroxyflavone*)



$C_{15}H_{10}O_6$ MW, 286

Yellow leaflets from MeOH. Does not melt below 340°. Sol. boiling MeOH, EtOH, AcOH. Spar. sol. other solvents. Insol. H_2O. KOH ⟶ reddish-yellow sol. Cold $NH_3.AgNO_3$ gives reddish-brown col. and is reduced on heating. Alc. $FeCl_3$ ⟶ reddish-brown col.

6 : 4'-*Di-Me ether*: $C_{17}H_{14}O_6$. MW, 314. Yellow cryst. M.p. 219°. *Diacetyl*: m.p. 149–50°.

7 : 4'-*Di-Me ether*: yellow plates. M.p. 211–13°.

5 : 7 : 4'-*Tri-Me ether*: $C_{18}H_{16}O_6$. MW, 328. Pale yellow plates from EtOH.Aq. M.p. 224–6°.

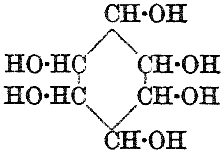
6 : 7 : 4'-*Tri-Me ether*: yellow microleaflets from AcOEt. M.p. 189–90°. Conc. $H_2SO_4 \longrightarrow$ greenish-yellow sol. *Acetyl*: microleaflets from AcOEt. M.p. 169°.

Tetra-Me ether: $C_{19}H_{18}O_6$. MW, 342. Exists in two forms. (i) Prisms from EtOH. M.p. 161°. (ii) Cryst. from EtOH. M.p. 142°.

Tetra-acetyl: needles from AcOEt or EtOH. M.p. 235–7°.

Bargellini, *Gazz. chim. ital.*, 1915, **45**, i, 77.
Goldschmiedt, Zerner, *Monatsh.*, 1910, **31**, 464.
Robinson, Schwarzenbach, *J. Chem. Soc.*, 1930, 829.
Wessely, Moser, *Monatsh.*, 1930, **56**, 97.
Sastri, Seshadri, *Proc. Indian Acad. Sci.*, 1946, 23A, 262.

Scyllitol (*Cocosite, cocositol, quercin, quercinite, hexahydroxycyclohexane, stereoisomer of inositol*)



$C_6H_{12}O_6$ MW, 180

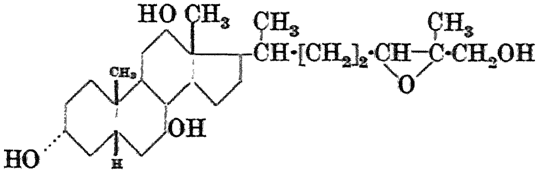
Occurs in dogfish (*Scyllium canicula*) and other fish; in *Acanthus vulgaris*, *Cornus florida*, Linn. (flowering dogwood) and various *Cocos* species. Prisms + $3H_2O$ from H_2O. M.p. 353° decomp. Spar. sol. H_2O. Insol. EtOH, Et_2O, MeOH, C_6H_6, $CHCl_3$.

Hexa-acetyl: m.p. 300°.

Needham, *Biochem. J.*, 1929, **23**, 319.
Hann, Sando, *J. Biol. Chem.*, 1926, **68**, 399.
Goodson, *J. Chem. Soc.*, 1920, **117**, 140.
Anderson, Wallis, *J. Am. Chem. Soc.*, 1948, **70**, 2931.

Scylloquercitol.
See under Quercitol.

Scymnol



Suggested structure

$C_{27}H_{46}O_5$ MW, 450

Occurs as sulphuric ester in bile of shark (*Scymnus borealis*). Cryst. + $2H_2O$ from Me_2CO.Aq., m.p. 115°: anhyd. cubes from AcOEt, m.p. 187°. $[\alpha]_D$ + 38·2° in EtOH.

Hydrochloride: two forms. (i) Needles from MeOH. M.p. 196°. (ii) M.p. 126°. Both forms give scymnol with alkalis.

Tetra-acetyl: plates from MeOH. M.p. 148°. B.p. 280°/0·005 mm.

Tschesche, *Z. physiol. Chem.*, 1931, **203**, 263.
Windaus, Bergmann, König, *Z. physiol. Chem.*, 1930, **189**, 148.
Oikawa, *Chem. Abstracts*, 1926, **20**, 401.
Asikari, *Chem. Zentr.*, 1939, II, 2666.
Bergmann, Pace, *J. Am. Chem. Soc.*, 1943, **65**, 477.

Sebacamic Acid.
See under Sebacic Acid.

Sebacanilic Acid (*Sebacic acid monoanilide*)

$$HOOC{\cdot}CH_2{\cdot}[CH_2]_6{\cdot}CH_2{\cdot}CO{\cdot}NHC_6H_5$$

$C_{16}H_{23}O_3N$ MW, 277

Leaflets from H_2O. M.p. 121–2°.

Me ester: $C_{17}H_{25}O_3N$. MW, 291. Needles from pet. ether. M.p. 67–8°.

Morgan, Walton, *J. Chem. Soc.*, 1936, 905.

Sebacic Acid (*Octane*-1 : 8-*dicarboxylic acid*)

$$HOOC{\cdot}CH_2{\cdot}[CH_2]_6{\cdot}CH_2{\cdot}COOH$$

$C_{10}H_{18}O_4$ MW, 202

Leaflets. M.p. 134·5° (132–3°). B.p. 294·5°/100 mm., 273°/50 mm., 243·5°/15 mm., 232°/10 mm. Sol. EtOH, Et_2O. Spar. sol. H_2O. Heat of comb. C_p 1297·3 Cal., C_v 1295·9 Cal. k (first) = $2{\cdot}6 \times 10^{-5}$ at 25°; (second) = $2{\cdot}6 \times 10^{-6}$ at 100°.

Me ester: $C_{11}H_{20}O_4$. MW, 216. M.p. 40–1°. B.p. 208°/20 mm. *Chloride*: $C_{11}H_{19}O_3Cl$. MW, 234·5. B.p. 177°/23 mm.

Di-Me ester: $C_{12}H_{22}O_4$. MW, 230. M.p. 26·4° (38°). B.p. 293°/754 mm., 175°/20 mm. D_4^{28} 0·98818. n_D^{28} 1·43549.

Et ester: $C_{12}H_{22}O_4$. MW, 230. M.p. 35°. B.p. 202–3°/15 mm. $k = 1{\cdot}43 \times 10^{-5}$ at 25°.

Di-Et ester: $C_{14}H_{26}O_4$. MW, 258. M.p. 1·25°. B.p. 306°/773 mm., 158–9°/7·5 mm., 142°/3 mm. D_4^{20} 0·96461. n_D^{20} 1·43589.

Dibutyl ester: $C_{18}H_{34}O_4$. MW, 314. B.p. 344–5°. D^{15} 0·9329.

Di-isoamyl ester: $C_{20}H_{38}O_4$. MW, 342. M.p. 18°. B.p. 202–3°/2–3 mm. D_4^{25} 0·9230.

Glycol ester: $C_{12}H_{20}O_4$. MW, 228. M.p. 79°.

Trimethylene glycol ester: $C_{13}H_{22}O_4$. MW, 242. M.p. 14–17°. B.p. 130–3°/2 mm. D_4^{20} 1·0747. n_D^{20} 1·4719.

Phenyl ester: $C_{16}H_{22}O_4$. MW, 278. M.p. 65–6°.

Dibenzyl ester: $C_{24}H_{30}O_4$. MW, 382. M.p. 28·3°. B.p. 257°/4 mm. D_4^{20} 1·0556. n_D^{20} 1·5171.

Di-p-nitrobenzyl ester: $C_{24}H_{28}O_8N_2$. MW, 472. M.p. 72–6°.

Dimenthyl ester: $C_{30}H_{54}O_4$. MW, 478. B.p. 256–8°/20 mm.

Diphenacyl ester: $C_{26}H_{28}O_4$. MW, 404. M.p. 80–4°.

Dichloride: $C_{10}H_{16}O_2Cl_2$. MW, 239. B.p. 185–95°/30 mm., 182°/16 mm. $n_D^{15 \cdot 5}$ 1·46836.

Monoamide: sebacamic acid. $C_{10}H_{19}O_3N$. MW, 201. M.p. 170°.

Diamide: $C_{10}H_{20}O_2N_2$. MW, 200. M.p. 210° (208°).

Dinitrile: octamethylene dicyanide, sebaconitrile. $C_{10}H_{16}N_2$. MW, 164. B.p. 199–200°/15 mm.

Anhydride: $C_{10}H_{16}O_3$. MW, 184. Cryst. from C_6H_6. M.p. 78–9°.

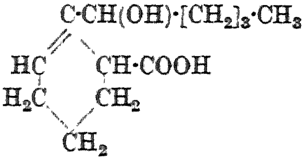
Monoanilide: *see* Sebacanilic Acid.

Dianilide: m.p. 198°.

Dihydrazide: m.p. 184–5°.

Industrial Chemist, 1952, 72.
Morgan, Walton, *J. Chem. Soc.*, 1936, 903.
Izard, U.S.P., 1,991,391, (*Chem. Abstracts*, 1935, **29**, 2176).
Spanagel, F.P., 796,410, (*Chem. Abstracts*, 1936, **30**, 6138).
Stoll, Rouvé, *Helv. Chim. Acta*, 1936, **19**, 253.
Fischl, Steiner, U.S.P., 1,876,652, (*Chem. Abstracts*, 1933, **27**, 102).
Naegeli, Münzel, D.R.P., 554,700, (*Chem. Abstracts*, 1932, **26**, 5970).
Landa, Kejvan, *Chem. Abstracts*, 1932, **26**, 78.
Montonna, *J. Am. Chem. Soc.*, 1927, **49**, 2114.
Boedtker, *Chem. Abstracts*, 1924, **18**, 3043.

Sedanolic Acid



$C_{12}H_{20}O_3$ MW, 212

Needles from C_6H_6. M.p. 88–9°. Sol. Et_2O, C_6H_6, hot pet. ether. Insol. H_2O.

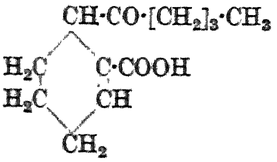
Lactone: sedanolide. $C_{12}H_{18}O_2$. MW, 194. Occurs in celery oil. B.p. 185°/17 mm. $D^{24 \cdot 5}$ 1·0383. $n_D^{24 \cdot 5}$ 1·4923. $[\alpha]_D^{26 \cdot 5} - 23 \cdot 66°$.

Ciamician, Silber, *Ber.*, 1897, **30**, 497.

Sedanolide.

See under Sedanolic Acid.

Sedanonic Acid



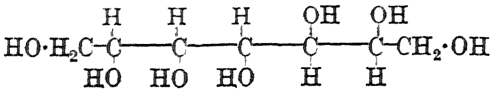
$C_{12}H_{18}O_3$ MW, 210

Saponification product from celery oil. Needles from C_6H_6. M.p. 113°. Sol. EtOH, AcOH. Mod. sol. Et_2O, C_6H_6. Spar. sol. pet. ether. Insol. H_2O.

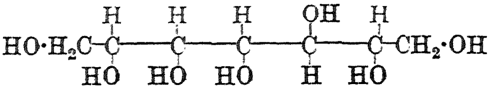
Oxime: m.p. 128°.

Ciamician, Silber, *Ber.*, 1897, **30**, 500.

Sedoheptitol



α-Form



β-Form

$C_7H_{16}O_7$ MW, 212

α-Form: Volemitol, *d*-β-Mannoheptitol.

Occurs in *Lactarius volemus*, etc. Needles from dil. EtOH. M.p. 154–5° (151–2°). $[\alpha]_D^{20}$ + 2·25° in H_2O. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O.

Triethylidene deriv.: cryst. M.p. 191–4°. $[\alpha]_D$ − 45·55° in $CHCl_3$, $[\alpha]_D^{20}$ − 72·35° in $CHCl_3$, − 117·6° in Py.

Tribenzylidene deriv.: needles. M.p. 214–15° (199–200°). $[\alpha]_D^{20}$ −1·7° in $CHCl_3$, − 48·4° in Py.

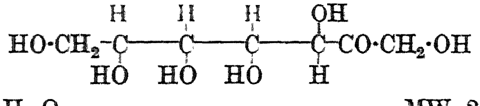
β-Form:

Plates or needles from dil. EtOH. M.p. 127–8°.

Tribenzylidene deriv.: m.p. 272·5°.

Bougault, Allard, *Compt. rend.*, 1902, **135**, 796.
La Forge, Hudson, *J. Biol. Chem.*, 1917, **30**, 68; 1928, **79**, 1.
La Forge, *J. Biol. Chem.*, 1920, **42**, 375.
Ettel, *Chem. Zentr.*, 1929, II, 714.
Ettel, *Chem. Abstracts*, 1933, **27**, 1617.

Sedoheptose



$C_7H_{14}O_7$ MW, 210

Occurs in *Sedum spectabile*. Syrup. Weakly dextrorotatory. Reduces Fehling's. NaHg → α- + β-sedoheptitol.

Phenylosazone: cryst. M.p. 197° decomp.

p-*Bromophenylosazone*: yellow needles. M.p. 227–8° decomp.

La Forge, *J. Biol. Chem.*, 1920, **42**, 367.
La Forge, Hudson, *J. Biol. Chem.*, 1917, **30**, 61.
Ettel, *Chem. Abstracts*, 1933, **27**, 1617.

Selacholeic Acid.

See Nervonic Acid.

Selachyl Alcohol (*Glycerol 1-octadecenyl ether, α-oleyl glyceryl ether*)

$CH_2 \cdot O \cdot [CH_2]_8 \cdot CH{:}CH \cdot [CH_2]_7 \cdot CH_3$
$CH \cdot OH$
$CH \cdot OH$

$C_{21}H_{42}O_3$ MW, 342

dl-.

Occurs in liver oil of *Centrophorus granulosis*. Cryst. from Me_2CO. M.p. 5·5–7·5°. B.p. 242°/5 mm., 145–6°/0·5 mm. D_4^{15} 0·9206. n_D^{15} 1·4690.

Diacetyl: syrup. B.p. 175–6°/0·005 mm. $n_D^{25.5}$ 1·4520.

p-*Toluenesulphonyl*: m.p. 18·5–19·5°. $n_D^{26.5}$ 1·4885.

Di-phenylurethane: m.p. 92–3°.

d-.

M.p. 48·5–49·5°. D^{25} 0·888. $[\alpha]_D^{55}$ − 4·35°.

Diacetyl: m.p. 18–19°. B.p. 170–2°/0·001 mm. $n_D^{24.5}$ 1·4521. $[\alpha]_D^{23}$ − 8·6°. D_4^{23} 0·937.

Di-phenylurethane: m.p. 93–4°. $[\alpha]_D^{24.5}$ − 6·0°.

l-.

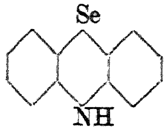
M.p. 48·5–49·5°.

Diacetyl: b.p. 182–3°/0·01 mm. D^{23} 0·935. $n_D^{23.5}$ 1·4530.

Di-phenylurethane: m.p. 93–4°. $[\alpha]_D^{23}$ + 5·8°.

Davies, Heilbron, Jones, *J. Chem. Soc.*, 1933, 165.
Baer, Rubin, Fischer, *J. Biol. Chem.*, 1944, **155**, 447.

Selenazine (*Phenoselenazine, selenodiphenylamine*)



$C_{12}H_9NSe$ MW, 246

Yellow leaflets from C_6H_6. M.p. 195°. Sol. ord. org. solvents. Oxidises in air.

N-*Acetyl*: m.p. 176°.

N-*Me*: $C_{13}H_{11}NSe$. MW, 260. M.p. 138–9°.

Cornelius, *Chem.-Ztg.*, 1913, **37**, 198.
Weizmann, Stephen, *Proc. Chem. Soc.*, 1913, **29**, 196.

Selenobenzoic Acid

$C_6H_5 \cdot COSeH$

C_7H_6OSe MW, 185

Red cryst. from MeOH. M.p. 133°.

p-*Tolyl ester*: $C_{14}H_{12}OSe$. MW, 275. Prisms from EtOH. M.p. 71–2°. Turns red in air.

p-*Methoxyphenyl ester*: $C_{14}H_{12}O_2Se$. MW, 291. Cryst. from EtOH. M.p. 97°.

p-*Ethoxyphenyl ester*: $C_{15}H_{14}O_2Se$. MW, 305. Needles from ligroin. M.p. 97°.

Amide: C_7H_7NSe. MW, 184. Golden needles from Et_2O. M.p. 126° (115°).

Mingoia, *Gazz. chim. ital.*, 1926, **56**, 835.
Becker, Meyer, *Ber.*, 1904, **37**, 2550.

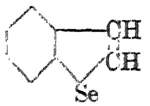
Selenodiphenylamine.

See Selenazine.

Selenofuran.

See Selenophene.

Selenonaphthene (*Benzselenophene*)



C_8H_6Se MW, 181

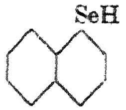
Leaflets from MeOH. M.p. 53–4° (50–1°). B.p. 238·5–240°/775 mm. Sol. ord. org. solvents.

Picrate: m.p. 156–7°.

Styphnate: m.p. 146–7°.

Komppa, Nyman, *J. prakt. Chem.*, 1934, **139**, 229.

1-Selenonaphthol

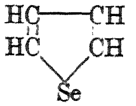


$C_{10}H_8Se$ MW, 207

Red liquid. B.p. 165–7°/20 mm.

Taboury, *Compt. rend.*, 1904, **138**, 982.

Selenophene (*Selenofuran*)



C_4H_4Se MW, 131

Oil. F.p. − 38°. B.p. 109·9–110·1°/752·1 mm. Sol. Me_2CO, C_6H_6, CS_2. Insol. H_2O. D_4^{15} 1·5301. n_D^{15} 1·568. Solvent for S. $H_2SO_4 \longrightarrow$ greenish-black mass.

McMahon, Pearson, Robinson, *J. Chem. Soc.*, 1933, 1644 (*Bibl.*).
Suginome, Umezawa, *Bull. Chem. Soc. Japan*, 1936, **11**, 157 (*Bibl.*).
Day, *Science Progress*, 1929, **23**, 585 (*Review*).
Mazza, Solazzo, *Chem. Abstracts*, 1929, **23**, 2417.

Selenophenol

$C_6H_5 \cdot SeH$

C_6H_6Se MW, 157

Oil. B.p. 183·6°/760 mm. Sol. Et_2O, CCl_4. Spar. sol. H_2O. D^{15} 1·4865. Oxidises in air to $(C_6H_5)_2Se_2$.

Lesser, Weiss, *Ber.*, 1914, **47**, 2522.
Bayer, D.R.P., 264,940, (*Chem. Abstracts*, 1914, **8**, 1351).
Foster, *Organic Syntheses*, 1944, XXIV, 89.

Selenourea

$H_2N \cdot CSe \cdot NH_2$

CH_4N_2Se MW, 123

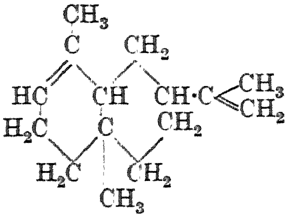
Prisms or needles from H_2O. M.p. 200° decomp. (slow heat). Mod. sol. H_2O. Spar. sol. EtOH, Et_2O.

Methiodide: yellow cryst. M.p. 187–8° decomp.

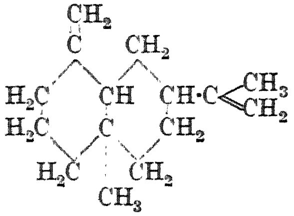
Methosulphate: yellow cryst. M.p. 67–70° decomp.

Verneuil, *Ann. chim.*, 1886, **9**, 292.

Selinene



α-.



β-.

$C_{15}H_{24}$ MW, 204

α-.

B.p. 268–72°, 128–32°/11 mm. D_{15}^{15} 0·9232. n^{20} 1·5048. $[\alpha]_D$ + 31°.

β-.

Occurs in celery oil. B.p. 121–2°/6 mm. D_{25}^{15} 0·9170. n_D^{21} 1·4956. $[\alpha]_D$ + 61°. Dihydrochloride + alkali ⟶ α-form.

Schimmel, *Chem. Zentr.*, 1910, I, 1719.
Ruzicka, Wind, Koolhaas, *Helv. Chim. Acta*, 1931, **14**, 1138.

Selinenol.

See Eudesmol.

Semicarbazide (*Hydrazinoformamide, carbazinamide*)

$$\underset{4}{NH_2}\cdot CO\cdot \underset{3\ \ 2}{NH}\cdot \underset{1}{NH_2}$$

CH_5ON_3 MW, 75

Prisms from EtOH. M.p. 96°. Reacts with aldehydes and ketones to give characteristic semicarbazones, R·CH:N·NH·CO·NH_2 or R_2C:N·NH·CO·NH_2.

B,HF: m.p. below 100° decomp.

B,HCl: m.p. 175°.

B,HNO_3: m.p. 123° decomp.

B,H_2SO_4: m.p. 145° decomp.

B_2,H_2SO_4: m.p. 143°.

B,$HClO_3$: explodes at 278°.

B,H·COOH: m.p. 128°.

B,$(COOH)_2$: m.p. 146° (133° decomp.).

Acid maleate: m.p. 133° decomp.

Maleate: m.p. 100°.

Picrate: m.p. 166° decomp.

1-*Acetyl*: m.p. 165°.

1-*Chloroacetyl*: m.p. 111–12°.

1-*Dichloroacetyl*: m.p. 108°.

1-*Trichloroacetyl*: m.p. 154° decomp.

1-*Benzoyl*: m.p. 225°.

Audrieth, *J. Am. Chem. Soc.*, 1930, **52**, 1250.
Mistry, Guha, *J. Indian Chem. Soc.*, 1930, I, 793.
Ingersoll, Bircher, Brubaker, *Organic Syntheses*, Collective Vol. I, 472.

3-Semicarbazidobenzamide.

See Kryogenin.

Seminose.

See Mannose.

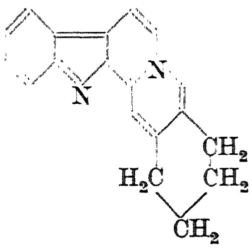
Semioxamazide.

See under Oxamic Acid.

Sempervine.

See Sempervirine.

Sempervirine (*Sempervine*)



$C_{19}H_{16}N_2$ MW, 272

Occurs in *Gelsemium sempervirens*, Ait. Cryst. + $1H_2O$, m.p. 258–60°: cryst. from $CHCl_3$, m.p. 223°: cryst. from EtOH, m.p. 254°. Sol. EtOH, $CHCl_3$, Py. Insol. Et_2O, C_6H_6.

Methochloride: m.p. 330–2°.

Methopicrate, m.p. 239–40°.

Hasenfratz, *Compt. rend.*, 1933, **196**, 1530.
Chou, *Chem. Abstracts*, 1931, **25**, 5736.
Stevenson, Sayre, *Chem. Abstracts*, 1916, **10**, 804.
Woodward, Witkop, *J. Am. Chem. Soc.*, 1949, **71**, 379.
Bentley, Stevens, *Nature*, 1949, **164**, 141.

Senecifoline

$C_{18}H_{27}O_8N$ MW, 385

Plates from $CHCl_3$–pet. ether. M.p. 194–5°. Sol. EtOH, Et_2O, $CHCl_3$. Insol. H_2O, pet. ether. $[\alpha]_D$ + 28·8° in EtOH.

B,HCl: m.p. 260° decomp. $[\alpha]_D$ − 20° in H_2O.

B,HI: m.p. 248° decomp.

B,HNO_3: m.p. 240° decomp.

B,$HAuCl_4$: m.p. 220° decomp.

Watt, *J. Chem. Soc.*, 1909, **95**, 469.

Senecifolinene.

See Retronecine.

Senecioaldehyde.

See 2 : 2-Dimethylacrolein.

Senecioic Acid.
See 2 : 2-Dimethylacrylic Acid.

Seneciophenone (*2-Benzoylisobutylene, ω-isopropylideneacetophenone, 4-keto-2-methyl-4-phenyl-2-butylene*)

$(CH_3)_2C{:}CH{\cdot}CO{\cdot}C_6H_5$

$C_{11}H_{12}O$ MW, 160

B.p. 134–6°/12 mm., 104–6°/5 mm. n_D^{23} 1·5519.

Dibromide : cryst. from MeOH. M.p. 78–9°.

Darzens, *Compt. rend.*, 1940, **211**, 435.

Seneciphylline

$C_{17}H_{23}O_5N$ MW, 321

Occurs in *Senecio platyphyllus*, D.C. Plates from EtOH. M.p. 217–18° decomp. Sol. $CHCl_3$. Mod. sol. EtOH, Me_2CO. Spar. sol. Et_2O, ligroin. $[\alpha]_D$ − 128·04° in $CHCl_3$.

Perchlorate : decomp. at 245°.
Aurichloride : m.p. 162–3° decomp.
Platinichloride : decomp. at 240°.
Picrate : m.p. 182–3°.
Methiodide : m.p. 231–2° decomp.

Orechoff, *Ber.*, 1935, **68**, 654.
Konovalova, Orekhov, *Bull. soc. chim.*, 1937, [5], **4**, 2037.

Sennite.
See under Inositol.

***p*-Septiphenyl.**
See p-Heptaphenyl.

Serine (*1-Aminohydracrylic acid, 2-hydroxy-1-aminopropionic acid, 2-hydroxy-α-alanine*)

$HO{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$

$C_3H_7O_3N$ MW, 105

Widely distributed in animal proteins.

d-.
Prisms from H_2O. M.p. 228° decomp. $[\alpha]_D^{20}$ + 6·87° in H_2O.

l-.
$[\alpha]_D^{20}$ − 6·83° in H_2O.
Me ester : *hydrochloride*, m.p. 167°.

dl-.
Leaflets from H_2O. M.p. 246° decomp. Sol. H_2O. Insol. EtOH, Et_2O. $H_2O_2 + FeSO_4 \longrightarrow HO{\cdot}CH_2{\cdot}CHO$. $FeCl_3 \longrightarrow$ red col.

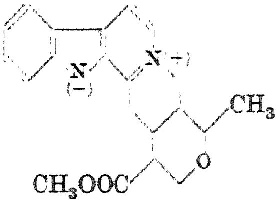
Me ester : $C_4H_9O_3N$. MW, 119. *B,HCl* : m.p. 114°.
Et ester : $C_5H_{11}O_3N$. MW 133. *B,HCl* : m.p. 100–2°.
N-*Chloroacetyl* : m.p. 122–3°.
N-1-*Bromopropionyl* : m.p. about 143°.
Benzoyl deriv. : decomp. at 149–50°. *B,HCl* : m.p. 185–6°. *Picrate* : m.p. 168–9°.
Phosphoric ester : serine-phosphoric acid. $C_3H_8O_6NP$. Ba salt forms white flocks in hot H_2O.
Sulphuric ester : $C_3H_7O_6NS$. M.p. 203–5° decomp.
Et ether : $C_5H_{11}O_3N$. MW, 133. Needles from EtOH. M.p. 256° decomp.
Picrolonate : decomp. at 265°.
Phenylurethane : m.p. 159°.

Leuchs, Geiger, *Ber.*, 1906, **39**, 2645.
Fischer, Jacobs, *ibid.*, 2944.
Mitra, *J. Indian Chem. Soc.*, 1930, **7**, 799.
Dunn, Redemann, Smith, *J. Biol. Chem.*, 1934, **104**, 511 (*Bibl.*).
Levene, Schormüller, *J. Biol. Chem.*, 1934, **106**, 595; **105**, 547.
Carter, West, *Organic Syntheses*, 1940, XX, 81.

Serine-phosphoric Acid.
See under Serine.

Serotonin.
See 5-Hydroxytryptamine.

Serpentine



Suggested structure

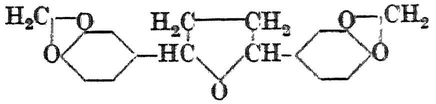
$C_{21}H_{22}O_3N_2$ MW, 350

Alkaloid from *Rauwolfia serpentina*, Benth. Cryst. from EtOH. M.p. 157°.

B,HCl : cryst. + H_2O. M.p. 246–8°. $[\alpha]_D^{23}$ + 178° in H_2O.
B,H₂SO₄ : m.p. 214°.
B,HNO₃ : m.p. 170–72°.
B,HClO₄ : m.p. 255–6°. $[\alpha]_D^{22}$ + 185° in Me_2CO.
Picrate : m.p. 261–2°.
Methiodide : m.p. 246°.

Schlittler, Schwarz, *Helv. Chim. Acta*, 1950, **33**, 1463.
Bader, Schwarz, *Helv. Chim. Acta*, 1952, **35**, 1594.

Sesamin

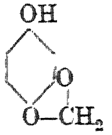


$C_{18}H_{16}O_5$ MW, 312

Occurs in sesame oil. Needles from EtOH. M.p. 123°. Sol. Me_2CO, $CHCl_3$. Spar. sol. Et_2O, pet. ether. $[\alpha]_D^{20}$ + 68·23° in $CHCl_3$.

Kreis, *Chem. Abstracts*, 1930, **24**, 2000.
Adriani, *Chem. Abstracts*, 1929, **23**, 2054.
Böeseken, Cohen, *Biochem. Z.*, 1928, **201**, 454.
Bertram, van der Steur, Waterman, *Biochem. Z.*, 1928, **197**, 1.

Sesamol (3 : 4-*Methylenedioxyphenol*, 1 : 2 : 4-*trihydroxybenzene* 1 : 2-*methylene ether*)



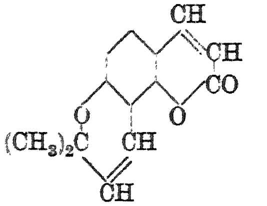
$C_7H_6O_3$ MW, 138

M.p. 65·8°. Sol. EtOH, Et_2O. Spar. sol. H_2O, pet. ether. $FeCl_3 \longrightarrow$ violet-brown col. Colours rapidly in air.

β-Glucoside: $C_{13}H_{16}O_8$. Cryst. + $1H_2O$. M.p. 168–9°.

Böeseken, Cohen, Kip, *Rec. trav. chim.*, 1936, **55**, 815.

Seselin

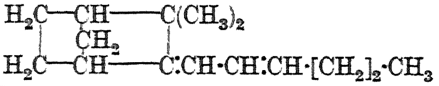


$C_{14}H_{12}O_3$ MW, 228

Constituent of fruit of *Selesi indicum* (Wall.) W. and A. Cryst. from MeOH–Et_2O. M.p. 119–20°. AcOH–$H_2SO_4 \longrightarrow$ umbelliferone.

Bose, Guha, *Chem. Zentr.*, 1937, II, 238.
Späth, Bose, Matzke, Guha, *Ber.*, 1939, **72**, 821.
Späth, Hillel, *ibid.*, 963, 2093.

Sesquicamphene



$C_{15}H_{24}$ MW, 204

Occurs in camphor oil. B.p. 255°, 140°/10 mm., 129–33°/8 mm. D^{20} 0·9015. n_D 1·50058. $[\alpha]_D$ + 73·5°.

Semmler, Rosenberg, *Ber.*, 1913, **46**, 768.
Langlois, *Ann. chim.*, 1919, **12**, 358.

Sesquicitronellene

$C_{15}H_{24}$ MW, 204

Occurs in Java citronella oil. B.p. 138–40°/9 mm. D^{20} 0·8489. n_D 1·53252.

Semmler, Spornitz, *Ber.*, 1913, **46**, 4025.

Sesquigoyol

$C_{15}H_{26}O$ MW, 222

Sesquiterpene alcohol from oil of *Pinus formosama*, Hayata. Said to be identical with γ-cardinol. M.p. 137–137·5°. B.p. 285–9°, 160–5°/8 mm. $[\alpha]_D^{20}$ + 99·5°, $[\alpha]_D$ + 93·4°.

Acetyl: b.p. 152–5°/4 mm. n_D^{32} 1·4902. $[\alpha]_D^{32}$ + 22·2°.

Yeigai, Sabe, *J. Chem. Soc. Japan*, 1935, **56**, 1118.
Sebe, *Chem. Abstracts*, 1943, **37**, 4064.

***p*-Sexiphenyl.**

See p-Hexaphenyl.

Shellolic Acid.

$C_{15}H_{20}O_6$ MW, 296

Isolated from shellac. Plates from H_2O. M.p. 206°. Sol. EtOH. Spar. sol. Et_2O, AcOEt, C_6H_6, $CHCl_3$. Insol. petrol.

A structural formula has been proposed for shellolic acid by Nagel and Martens, but Kirk, Spoerri and Gardner have put forward evidence which indicates that the proposed structure is incorrect.

Di-Me ester: $C_{17}H_{24}O_6$. MW, 324. Prisms from EtOH. M.p. 156°. $[\alpha]_D^{20}$ + 36°.

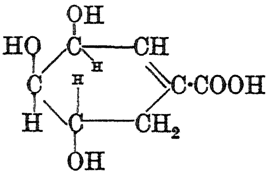
Di-hydrazide: prisms from H_2O. M.p. 246° decomp.

Di-phenylurethane: cryst. from CCl_4. M.p. about 92–4°.

Dihydro deriv.: m.p. 157–8°.

Kirk, Spoerri, Gardner, *J. Am. Chem. Soc.*, 1941, **63**, 1243.
Nagel, Martens, *Ber.*, 1937, **70**, 2173.
Harries, Nagel, *Ber.*, 1922, **55**, 3833.

Shikimic Acid



$C_7H_{10}O_5$ MW, 174

Constituent of fruits of *Illicium religiosum*, Siebold. Cryst. from H_2O. M.p. 178–80°. Insol. EtOH, Et_2O, $CHCl_3$.

A,CH_3NH_2: m.p. 163–4°.
A,$NH_2{\cdot}NH_2$: m.p. 147–8°.
A,$C_6H_5NH_2$: m.p. 194–5°.
A,Py: micro-plates. M.p. 184–5°.
A,Quinine: needles from EtOH. M.p. 221–2°.
A,Strychnine: plates. Sinters at 154°. M.p. 234–6°.

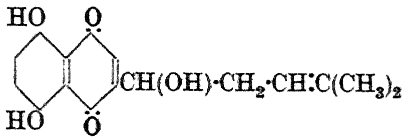
Me ester: needles from AcOH–petrol. M.p. 113–14°.

Triacetyl: b.p. 200–10°/1 mm.

Me_2CO comp.: cryst. from AcOH–petrol. M.p. 184°.

Fischer, Dangschat, *Naturwissenschaften*, 1938, **26**, 562; *Helv. Chim. Acta*, 1937, **20**, 705; 1935, **18**, 1206; 1934, **17**, 1200.

Shikonin (d-*Alkannin*)



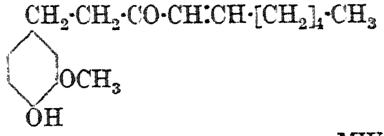
$C_{16}H_{16}O_5$ MW, 288

Occurs in root of *Lithospermum erythrorhizon*, (" shikon "). Brownish-red needles from C_6H_6. M.p. 143°. Reduces Tollen's. $FeCl_3 \longrightarrow$ indigo-blue ppt.

Me ether: $C_{17}H_{18}O_5$. MW, 302. M.p. 105°.

Karoda, Wada, *Chem. Zentr.*, 1937, I, 3156.

Shogaol



$C_{17}H_{24}O_3$ MW, 276

Occurs in ginger (" shoga "). Pale yellow oil. B.p. 201·3°/2–2·5 mm. Sol. EtOH, AcOH. D_4^{25} 1·0419. n_D^{25} 1·52518. $FeCl_3 \longrightarrow$ green col. Reduces Tollen's.

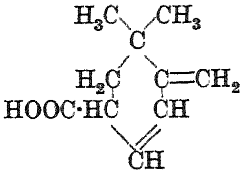
Acetyl deriv.: b.p. 183–8°/0·6 mm.

Me ether: $C_{18}H_{26}O_3$. MW, 290. Yellow oil. B.p. 160–5°/0·06 mm.

Et ether: $C_{19}H_{28}O_3$. MW, 304. Yellow oil. B.p. 181–6°/0·65 mm.

Nomura, Iwamoto, Murakami, *Chem. Abstracts*, 1930, **24**, 2445.

Shonanic Acid



$C_{10}H_{14}O_2$ MW, 166

Occurs in wood of *Libocedrus formosana*, Florin. Cryst. from pet. ether. M.p. 40–1°. B.p. 264°/754 mm., 134–134·5°/6 mm. D_4^{46} 1·016. n_D^{46} 1·4842. $[\alpha]_D^{18}$ − 0·75° in EtOH.

Me ester: $C_{11}H_{16}O_2$. MW, 180. B.p. 222°, 113–14°/20 mm. D_4^{20} 0·9848. n_D^{20} 1·4758. $[\alpha]_D^{28}$ − 2·84°.

Et ester: $C_{12}H_{18}O_2$. MW, 194. B.p. 228–9°/759 mm., 106–8°/7 mm. D_4^{20} 0·9568. n_D^{20} 1·4674. $[\alpha]_D^{18}$ − 4·24°.

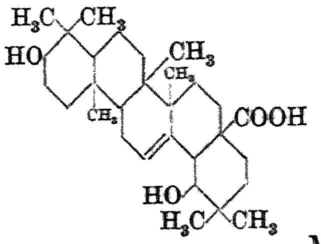
Chloride: $C_{10}H_{13}OCl$. MW, 184·5. B.p. 215°, 106–7°/20 mm. D_4^{20} 1·0577. n_D^{20} 1·4955.

Amide: $C_{10}H_{15}ON$. MW, 165. M.p. 116–17°.

Anilide: m.p. 111–12°.

Ichikawa, *Bull. Chem. Soc. Japan*, 1936, **11**, 759; 1937, **12**, 253, 258, 267.

Siaresinolic Acid



$C_{30}H_{48}O_4$ MW, 472

Isolated from Siam benzoin (Siamese gum). Plates from MeOH–Et_2O. M.p. 274–5°. $[\alpha]_D^{24}$ + 98·5° in $CHCl_3$.

Me ester: prismatic plates from petrol. M.p. 180–1°. $[\alpha]_D^{24}$ + 48·4° in $CHCl_3$. *Acetyl*: needles from MeOH. M.p. 110–20°.

Et ester: needles or plates from petrol. M.p. 108°.

Bromolactone: m.p. 178–80°. $[\alpha]_D^{24}$ + 151·8° in $CHCl_3$.

Winterstein, Egli, *Z. physiol. Chem.*, 1931, **202**, 207.

Zinke, Lieb, *Monatsh.*, 1918, **39**, 95, 627.

Bilham, Kon, Ross, *J. Chem. Soc.*, 1942, 540.

Ruzicka, Grob, Egli, Jeger, *Helv. Chim. Acta*, 1943, **26**, 1218.

Barton, Holness, *J. Chem. Soc.*, 1952, 78.

Silicon tetraethyl (*Tetraethylsilane*)

$Si(C_2H_5)_4$

$C_8H_{20}Si$ MW, 144

B.p. 152·8–153·2°/758·5 mm. D_4^{25} 0·7620. $n_D^{25\cdot1}$ 1·4246.

Kipping, Lloyd, *J. Chem. Soc.*, 1901, **79**, 456.

Bygden, *Ber.*, 1911, **44**, 2650.

Larsson, *Chem. Abstracts*, 1949, **43**, 2928.

Silicon tetramethyl (*Tetramethylsilane*)

$Si(CH_3)_4$

$C_4H_{12}Si$ MW, 88

Mod. stable. B.p. 26·6°. Two forms of solid: α (unstable), m.p. −102·2°; β, m.p. −99·1°.

Aston, Kennedy, Messerly, *J. Am. Chem. Soc.*, 1941, **63**, 2343.

Silicon tetraphenyl (*Tetraphenylsilane*)

$Si(C_6H_5)_4$

$C_{24}H_{20}Si$ MW, 336

Cryst. from Et_2O. M.p. 237·5°. B.p. 530°.

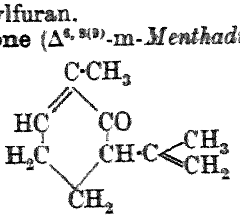
Polis, *Ber.*, 1885, **18**, 1540.

Kipping, Blackburn, *J. Chem. Soc.*, 1935, 1085.

Silvan.

See 2-Methylfuran.

Silvecarvone ($\Delta^{6,8(9)}$-m-*Menthadienone*-2)



$C_{10}H_{14}O$ MW, 150

Oil.

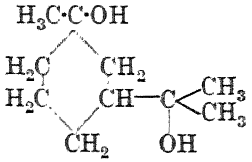
Semicarbazone: m.p. 175–7°.

Wallach, *Ann.*, 1907, **357**, 74.

Silver Salt.

See under Anthraquinone-2-sulphonic Acid.

Silveterpin (d-m-*Menthandiol*-1 : 8)



$C_{10}H_{20}O_2$ MW, 172

α-.

M.p. 137–8°. $[\alpha]_D$ + 27·74° in $CHCl_3$. Sublimes.

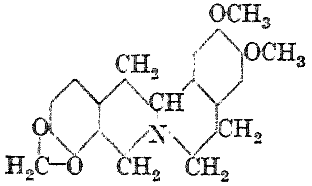
β-.

M.p. 70–5°. $[\alpha]_D$ + 20·93° in $CHCl_3$.

Synthetic : needles from Et_2O–petrol. M.p. 91–2°.

Haworth, Perkin, Wallach, *J. Chem. Soc.*, 1913, **103**, 1233; *Ann.*, 1913, **399**, 161.
Alder, Vogt, *Ann.*, 1949, **564**, 79.

Sinactine (*Tetrahydroepiberberine*)



$C_{20}H_{21}O_4N$ MW, 339

l-.

Occurs in *Sinomenium acutum*. Prisms from EtOH. M.p. 175°. Sol. $CHCl_3$. Spar. sol. MeOH, EtOH. Insol. H_2O. $[\alpha]_D$ − 312° in $CHCl_3$.

B,HCl : m.p. about 272° decomp.
B_2,H_2PtCl_6 : m.p. 245–7°.

dl-.

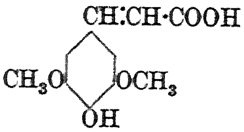
Needles from EtOH. M.p. 168°.
B,HCl : m.p. about 286° decomp.

Goto, Kitasato, *J. Chem. Soc.*, 1930, 1234.

Sinamin.

See Triallyltricyanamide.

Sinapic Acid (*Sinapinic acid, 4-hydroxy*-3 : 5-*dimethoxycinnamic acid*)



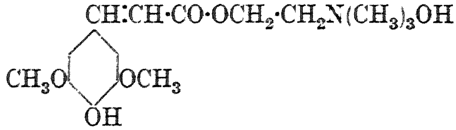
$C_{11}H_{12}O_5$ MW, 224

Yellow needles from EtOH. M.p. 192°. Sol. hot EtOH. Spar. sol. H_2O, Et_2O. Ox. ⟶ 2 : 6-dimethoxy-*p*-benzoquinone. $FeCl_3$ ⟶ red col.

Carbethoxyl : m.p. 174°.
Acetyl : m.p. 188–93°. *Chloride* : m.p. 142–4°.
Me ester : $C_{12}H_{14}O_5$. MW, 238. Cryst. from MeOH.Aq. M.p. 91–2°.
Et ester : $C_{13}H_{16}O_5$. MW, 252. Cryst. + $1H_2O$ from EtOH.Aq. M.p. 80–1°.
Choline ester : *see* Sinapin.
4-*Me ether* : 3 : 4 : 5-trimethoxycinnamic acid. $C_{12}H_{14}O_5$. MW, 238. Needles from H_2O. M.p. 123·5–124·5° (126·8°). Sol. hot H_2O. *Me ester* : $C_{13}H_{16}O_5$. MW, 252. Yellow leaflets from EtOH.Aq. M.p. 91–91·5°.

Späth, *Monatsh.*, 1920, **41**, 271.
Mauthner, *Ber.*, 1908, **41**, 2531.
Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 519.
Chmielewska, *Chem. Zentr.*, 1937, I, 3156.

Sinapin (4-*Hydroxy*-3 : 5-*dimethoxycinnamic acid choline ester*)



$C_{16}H_{25}O_6N$ MW, 327

Occurs in black mustard seeds. Unstable. Hyd. ⟶ sinapic acid + choline.

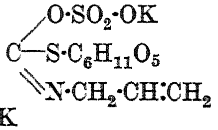
Salts ($B = C_{16}H_{23}O_5N$) :—
B,HBr : m.p. anhyd. 107–15°.
B,HI : m.p. 185–6°.
B,H_2SO_4 : m.p. anhyd. 126·5–127·5°.

Späth, *Monatsh.*, 1920, **41**, 271.

Sincalin.

See Choline.

Sinigrin (*Sinigroside, potassium myronate*)



$C_{10}H_{16}O_9NS_2K$ MW, 397

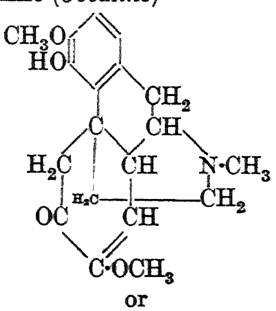
Occurs in black mustard seeds and *Alliaria officinalis*, D.C. M.p. 127–9°. $[\alpha]_D^{18}$ − 16·4° in H_2O.

Benik, Brauss, *Z. physiol. Chem.*, 1923, **126**, 210.
Herissey, Boivin, *Chem. Abstracts*, 1928, **22**, 667.
Schneider, Fischer, Specht, *Ber.*, 1930, **63**, 2789.

Sinigroside.

See Sinigrin.

Sinomenine (*Coculine*)



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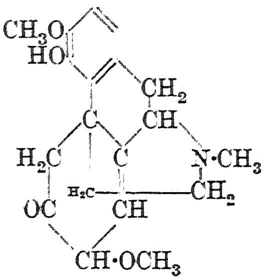


$C_{19}H_{23}O_4N$ MW, 329

Occurs in *Sinomenium acutum*. Needles from C_6H_6. M.p. 162°. Sol. EtOH, $CHCl_3$, Me_2CO, amyl alcohol. Mod. sol. Et_2O, C_6H_6. Spar. sol. H_2O. Insol. pet. ether. $[\alpha]_D^{26}$ − 70·70°.
B,HCl: m.p. 231°.
B,HBr: m.p. 231°.
B,HI: m.p. 233°.
B,HNO₃: m.p. 215° decomp.
Oxime: m.p. 233°.
Picrate: m.p. about 140°.
Methiodide: decomp. at 255°.

Goto, Michinaki, Shishido, *Ann.*, 1935, **515**, 297.

Sinomenol.
See under 2 : 3 : 5 : 6-Tetrahydroxyphenanthrene.

Sitostane.
See Stigmastane.

γ-Sitostane

$C_{29}H_{52}(C_{28}H_{50})$ MW, 400 (386)

Derived from γ-sitosterol. M.p. 87°. $[\alpha]_D^{15}$ + 20·2° in $CHCl_3$.

Bonstedt, *Z. physiol. Chem.*, 1928, **176**, 279.

Sitostanol.
See Stigmastanol.

α₁-Sitostanol

$C_{29}H_{52}O$ MW, 416

M.p. 185–6°. $[\alpha]_D^{20}$ + 27°.
Acetyl: m.p. 147–8°. $[\alpha]_D^{19}$ + 39·4°.

Bernstein, Wallis, *J. Am. Chem. Soc.*, 1939, **61**, 2308.

γ-Sitostanol

$C_{29}H_{52}O(C_{28}H_{50}O)$ MW, 416 (402)

Plates from EtOH. M.p. 143–4°. $[\alpha]_D^{16\cdot5}$ + 20·8° in $CHCl_3$.
Acetyl: m.p. 144–5°. $[\alpha]_D^{20}$ + 12·4° in $CHCl_3$.

Bonstedt, *Z. physiol. Chem.*, 1928, **176**, 278.

Sitosterol

The phytosterol originally termed sitosterol is a complex mixture separated only with difficulty. Hence the purity of the following fractions must be accepted with reserve.

Cf. Barton, *J. Chem. Soc.*, 1945, 813, 818; 1946, 512, 517.

α₁-.

$C_{29}H_{48}O$ MW, 412

Widely distributed in plants in small amounts. Needles from EtOH. M.p. 164–6°. $[\alpha]_D^{28}$ − 1·7° in $CHCl_3$. Precipitated by digitonin.
Acetyl: plates from EtOH. M.p. 137°. $[\alpha]_D^{28}$ + 28·6° in $CHCl_3$.
Benzoyl: needles from EtOH–C_6H_6. M.p. 168–72°. $[\alpha]_D^{27}$ − 41·8° in $CHCl_3$.
3 : 5-*Dinitrobenzoyl*: plates from AcOEt. M.p. 222°. $[\alpha]_D^{24}$ − 37·2° in $CHCl_3$.

Wallis, Fernholz, *J. Am. Chem. Soc.*, 1936, **58**, 2446.
Bernstein, Wallis, *J. Am. Chem. Soc.*, 1939, **61**, 2308.
Bernstein, Wilson, Wallis, *J. Org. Chem.*, 1942, **7**, 103.

α₂-.

$C_{30}H_{50}O$ MW, 426

Widely distributed in plants in small amounts. Cryst. from EtOH–pet. ether. M.p. 156°. $[\alpha]_D^{25}$ + 3·5° in $CHCl_3$.
Acetyl: plates from EtOH. M.p. 124–6°. $[\alpha]_D^{27}$ + 16·5° in $CHCl_3$. Spar. sol. MeOH.
Benzoyl: needles from EtOH–C_6H_6. M.p. 164–6°. $[\alpha]_D^{26}$ + 27·4° in $CHCl_3$. Spar. sol. EtOH.
3 : 5-*Dinitrobenzoyl*: needles from Me_2CO. M.p. 206°. $[\alpha]_D^{20}$ + 26·4° in $CHCl_3$.

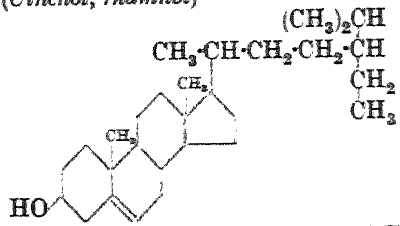
Wallis, Fernholz, *J. Am. Chem. Soc.*, 1936, **58**, 2446.

α₃-.

M.p. 142–3°. $[\alpha]_D^{20}$ + 5·2°.
Acetyl: m.p. 152–3°. $[\alpha]_D^{20}$ + 6·1°.
Benzoyl: m.p. 173–5°. $[\alpha]_D^{20}$ + 1·2°.
3 : 5-*Dinitrobenzoyl*: m.p. 210°. $[\alpha]_D^{20}$ + 12·2°.

Wallis, Fernholz, *J. Am. Chem. Soc.*, 1936, **58**, 2446.
Bernstein, Wallis, *J. Am. Chem. Soc.*, 1939, **61**, 1903.

β-. (*Cinchol, rhamnol*)



$C_{29}H_{50}O$ MW, 414

Widely distributed in plants. Needles from MeOH. M.p. 136–7°. $[\alpha]_D^{22}$ − 35° in $CHCl_3$.
Acetyl: needles from MeOH. M.p. 134° (128°). $[\alpha]_D^{20}$ − 38·5° in $CHCl_3$.
Benzoyl: m.p. 145·5°.

3 : 5-*Dinitrobenzoyl* : yellow cryst. from $CHCl_3$–MeOH. M.p. 202–4°. $[\alpha]_D^{22}$ − 10·6° in $CHCl_3$.

Anderson, Shriner, Burr, *J. Am. Chem. Soc.*, 1926, **48**, 2987.
Ichiba, *Chem. Zentr.*, 1936, I, 1027.
Windaus, Deppe, *Ber.*, 1933, **66**, 1254, 1689.
Dirscherl, *Z. physiol. Chem.*, 1938, **257**, 239.
Dirscherl, Nahm, *Ann.*, 1943, **555**, 57; 1947, **558**, 231.

γ-.

$C_{29}H_{50}O (C_{28}H_{48}O)$ MW, 414 (400)

Plates from EtOH. M.p. 147–8°. $[\alpha]_D$ − 43·13° in $CHCl_3$.
Acetyl : cryst. from EtOH. M.p. 143°. $[\alpha]_D$ − 47·7° in $CHCl_3$. *Dibromide* : needles from C_6H_6–MeOH. M.p. 140–1°. $[\alpha]_D$ − 46·23° in $CHCl_3$.
Benzoyl : m.p. 152°. $[\alpha]_D$ − 19·63° in $CHCl_3$.

Ichiba, *Chem. Zentr.*, 1936, I, 1027.
Sandqvist, Bengtsson, *Ber.*, 1931, **64**, 2167.

δ-.

$C_{29}H_{50}O$ MW, 414

Cryst. from EtOH. M.p. 146–7°. $[\alpha]_D$ − 23·9° in $CHCl_3$.
Acetyl : m.p. 115°. $[\alpha]_D$ − 24·35° in $CHCl_3$.
Benzoyl : m.p. 157–8°. $[\alpha]_D$ − 15·98° in $CHCl_3$.

Ichiba, *Chem. Zentr.*, 1936, I, 1027.

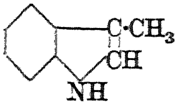
ε-.

$C_{29}H_{50}O$ MW, 414

M.p. 143–4°. $[\alpha]_D^{17}$ − 38·7° in $CHCl_3$.
Acetyl : plates from EtOH. M.p. 127–8°. $[\alpha]_D^{17}$ − 44·7° in $CHCl_3$.
3 : 5-*Dinitrobenzoyl* : m.p. 215–17°. $[\alpha]_D^{17}$ − 10° in $CHCl_3$.

Simpson, *J. Chem. Soc.*, 1927, 737.

Skatole (*Scatole*, *3-methylindole*, *β-methylindole*)

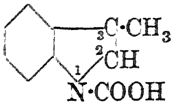


C_9H_9N MW, 131

Occurs in coal tar, beetroot, nectandra wood and fæces. Leaflets from ligroin. M.p. 95°. B.p. 265–6°/755 mm. $K_4Fe(CN)_6 + H_2SO_4 \longrightarrow$ violet col.
B_2,HCl : m.p. 167–8°.
N-Acetyl : m.p. 68°.
N-Propionyl : m.p. 45°.
Picrate : m.p. 170–1° decomp.
N-Me : *see* 1 : 3-Dimethylindole.

Kruber, D.R.P., 515,543, (*Chem. Abstracts*, 1931, **25**, 2443).
King, l'Ecuyer, *J. Chem. Soc.*, 1934, 1903.
Treffler, *Chem. Abstracts*, 1945, **39**, 1639.
Marion, Oldfield, *Can. J. Research*, 1947, **25**B, 1.

Skatole-1-carboxylic Acid (*3-Methylindole-N-carboxylic acid*)



$C_{10}H_9O_2N$ MW, 175

Occurs in beet. Needles. M.p. 162·5°. Sol. EtOH, Et_2O. Spar. sol. H_2O, C_6H_6.

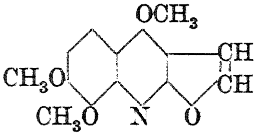
Lippmann, *Ber.*, 1924, **57**, 257.

Skatole-2-carboxylic Acid (*3-Methylindole-2-carboxylic acid*).

Needles from AcOH. M.p. 164–5° (167°). Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O, ligroin. $k = 4·7 \times 10^{-6}$ at 25°.
Me ester : cryst. from MeOH. M.p. 148°.
Et ester : needles from EtOH. M.p. 133–4°. Sol. Et_2O, C_6H_6.
Amide : needles from C_6H_6. M.p. 115°.
N-Me : cryst. from C_6H_6–EtOH. Decomp. about 213°. Sol. EtOH. Spar. sol. C_6H_6, pet. ether.

Perkin, Kermack, Robinson, *J. Chem. Soc.*, 1921, **119**, 1634.
Oddo, *Gazz. chim. ital.*, 1912, **42**, i, 370.

Skimmianine (*β-Fagarine*)

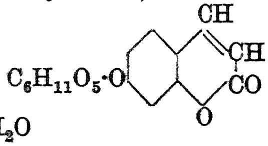


$C_{14}H_{13}O_4N$ MW, 259

Alkaloid from *Skimmia japonica*. M.p. 176°. H_2SO_4 + formaldehyde $\longrightarrow$ yellow col. changing to malachite green on warming.
Picrate : m.p. 197–8°.

Deulofeu, Labriola, Langhe, *J. Am. Chem. Soc.*, 1942, **64**, 2326.

Skimmin (*7-Hydroxycoumarin* d-*glucoside*, *umbelliferone* d-*glucoside*)



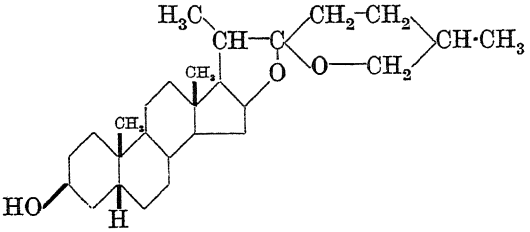
$C_{15}H_{16}O_8 \cdot H_2O$ MW, 342

Isolated from wood of *Skimmia japonica*. M.p. 219–21°. $[\alpha]_D^{18}$ − 79·8° in Py. 4% H_2SO_4 $\longrightarrow$ umbelliferone + glucose.
Tetra-acetyl : cryst. from MeOH. M.p. 183–4°. $[\alpha]_D^{16}$ − 63·3° in Py.

Späth, Neuefeld, *Rec. trav. chim.*, 1938, **57**, 535.

Skimmiol.
See Taraxerol.

Smilagenin (*Isosarsasapogenin*)



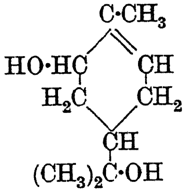
$C_{27}H_{44}O_3$ MW, 416

Occurs in Jamaica sarsaparilla root. Needles from Me_2CO. M.p. 183–4°. $[\alpha]_D^{25}$ − 69° in $CHCl_3$.

Acetyl deriv.: m.p. 150–1°. $[\alpha]_D^{25}$ − 59·6°.
Benzoyl deriv.: m.p. 181–181·5°.
o-*Bromobenzoyl deriv.*: m.p. 196–7°.

Farmer, Kon, *J. Chem. Soc.*, 1937, 414.
Askew, Farmer, Kon, *J. Chem. Soc.*, 1936, 1402.
Kon, Soper, Woolman, *J. Chem. Soc.*, 1939, 1201.
Marker *et al.*, *J. Am. Chem. Soc.*, 1940, **62**, 648.

Sobrerol (Δ^1-p-*Menthenediol*-6 : 8, 1-*methyl*-4-α-*hydroxyisopropylcyclohexenol*-6)



$C_{10}H_{18}O_2$ MW, 170

d-.
Prisms from H_2O, plates from EtOH. M.p. 150° (148–9°). $[\alpha]_D$ + 141° 16′ in EtOH.

l-.
Yellow cryst. M.p. 147–8°.

Dupont, *Chem. Abstracts*, 1922, **16**, 4340.
Gildemeister, Köhler, *Chem. Zentr.*, 1909, II, 2159.
Prilezhaev, Vershuk, *Chem. Abstracts*, 1930, **24**, 607.
Wallach, *Ann.*, 1917, **414**, 196.

Sodium methyl

CH_3Na MW, 38

Prepared from mercury dimethyl and sodium in an atmosphere of N_2. Burns explosively in air. Reacts with C_6H_6 giving sodium phenyl.

Schlenk, Holtz, *Ber.*, 1917, **50**, 266.

Sodium phenyl

C_6H_5Na MW, 100

Prepared from mercury diphenyl and sodium. Burns explosively in air.

Schlenk, Holtz, *Ber.*, 1917, **50**, 262.

Solancarpidine.
See Solasodine.

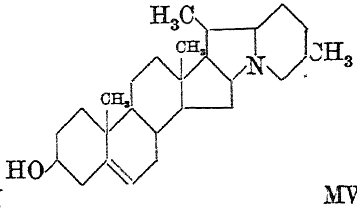
Solanidine-S

$C_{18}H_{31}ON$ MW, 277

Occurs in *Solanum sodomæum*, Linn. Needles. M.p. 249–53°.

Rochelmeyer, *Chem. Zentr.*, 1937, I, 1947.
Oddo, Caronna, *Ber.*, 1936, **69**, 283.

Solanidine-T



$C_{27}H_{43}ON$ MW, 397

Occurs in *Solanum tuberosum*, Linn. Cryst. from EtOH. M.p. 219° (216°). $[\alpha]_D$ − 27° in $CHCl_3$.

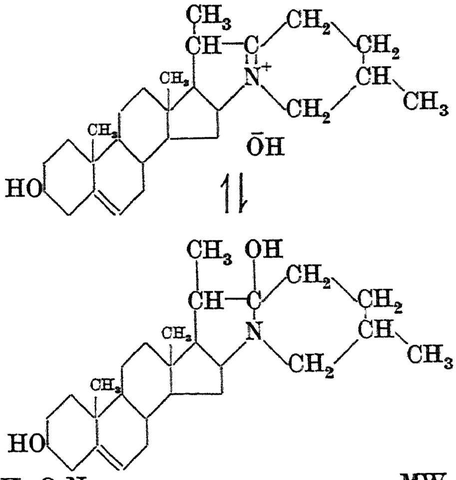
Acetyl deriv.: m.p. 204°.

Clemo, Morgan, Raper, *J. Chem. Soc.*, 1936, 1299.
Soltys, Wallenfels, *Ber.*, 1936, **69**, 811.
Heiduschka, Philippi, *Ber.*, 1935, **68**, 669.
Oddo, Caronna, *Ber.*, 1934, **67**, 451.
Dieterle, Rochelmeyer, *Arch. Pharm.*, 1935, **273**, 532.
Prelog, Szpilfogel, *Helv. Chim. Acta*, 1944, **27**, 390.
Uhle, Jacobs, *J. Biol. Chem.*, 1945, **160**, 243.

Solanine-D.
See Demissidine.

Solanine-S.
See Solasonine.

Solasodine (*Solancarpidine*)



$C_{27}H_{43}O_2N$ MW, 413

Occurs as glycoside in *Solanum xanthocarpum* and *S. aviculare*. M.p. 202°. $[\alpha]_D^{20}$ − 92·4° in C_6H_6. Gives usual sterol colour reactions.

Monoacetyl: plates from EtOH–AcOEt. M.p. 195°.

Monobenzoyl: m.p. 216–17°.

3 : 5-*Dinitrobenzoyl*: yellowish needles from EtOH. M.p. 191·5–193°.

Hydriodide: prisms from MeOH–Et_2O. M.p. 286° decomp.

Rochelmeyer, Chen, *Arch. Pharm.*, 1939, **277**, 329.
Briggs, *J. Chem. Soc.*, 1942, 3.
Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 600.

Solasonine (*Solanine-S, purapurine*)

$C_{45}H_{73}O_{16}N$ MW, 883

Glycoside isolated from *Solanum aviculare*. Sol. hot alcohols ⟶ gels on cooling. M.p. 279°. $[\alpha]_D$ − 69° in EtOH. Hyd. ⟶ solasodine, glucose, galactose and rhamnose. Conc. H_2SO_4 finally ⟶ crimson col. with brown fluor.

Briggs, *Nature*, 1939, **144**, 247; *J. Chem. Soc.*, 1942, 3.
Levi, *J. Soc. Chem. Ind.*, 1930, **49**, 395T.
Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 600.

Solvarsin.
See under 2-Aminophenol-4-arsonic Acid.

Soneryl.
See Neonal.

Sophocarpidine.
See Matrine.

Sophocarpine

$C_{15}H_{24}ON_2$ MW, 248

Cryst. + $1H_2O$ from H_2O. M.p. 81–2°, anhyd. 54–5°. $[\alpha]_D^{18}$ − 29·44° in EtOH.

B,HI: cryst. from EtOH. M.p. 277–9°.

Aurichloride: m.p. 166–7°.

Platinochloride: m.p. 209–12° decomp.

Methiodide: needles from EtOH. M.p. 200–2°.

Picrate: m.p. 155–7°.

Orechoff, Proskurnina, *Ber.*, 1934, **67**, 77.

Sophorabiose

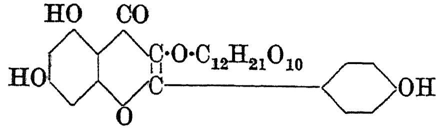
$C_{12}H_{22}O_{10}$ MW, 326

Disaccharide. Occurs as glycoside in the fruit of *Sophora japonica*, Linn. Hyd. ⟶ α-glucose + *l*-rhamnose.

Acetyl deriv.: $[\alpha]_D^{20}$ − 72·4° in H_2O.

Zemplén, Bognár, *Ber.*, 1942, **75**, 482.

Sophoraflavanoloside



$C_{27}H_{30}O_{16}$ MW, 610

Glycoside from green fruits of *Sophora japonica*. Yellow needles. M.p. 207–8°. $[\alpha]_D^{20}$ −61° in EtOH. Hyd. ⟶ kæmpferol + sophorose, m.p. 195–6°. Emulsin ⟶ *d*-glucose.

Rabaté, Dussy, *Bull. soc. chim. biol.*, 1938, **20**, 459.

Sophoramine

$C_{15}H_{20}ON_2$ MW, 244

M.p. 164–5°. $[\alpha]_D$ − 90·85°.

B,HCl: m.p. 247–8° decomp.

B,HI: m.p. 294–6°.

Aurichloride: m.p. 183–4° decomp.

Platinochloride: m.p. 245–7° decomp.

Picrate: m.p. 229–31° decomp.

Picrolonate: m.p. 173–5° decomp.

Orechoff, *Chem. Zentr.*, 1935, II, 2215.
Orechoff, Proskurnina, Konowalowa, *Ber.*, 1935, **68**, 431.

Sophoretin.
See Quercetin.

Sophoricol.
See Genistein.

Sophoricoside (*Genistein* β-*glucoside*)

$C_{21}H_{20}O_{10}$ MW, 432

Isolated from green pods of *Sophora japonica*. Prisms from EtOH. M.p. 297°. $[\alpha]_D^{20}$ − 46·7°. Acid hyd. ⟶ genistein + glucose.

Hexa-acetyl: cryst. from AcOH or EtOH. M.p. 230°.

Charaux, Rabaté, *Bull. soc. chim. biol.*, 1938, **20**, 454; *J. pharm. chim.*, 1935, **21**, 546.

Sophoridine

$C_{15}H_{26}ON_2$ MW, 250

Needles from pet. ether. M.p. 109–10°. $[\alpha]_D$ − 63·57°.

Aurichloride: m.p. 189–90°.

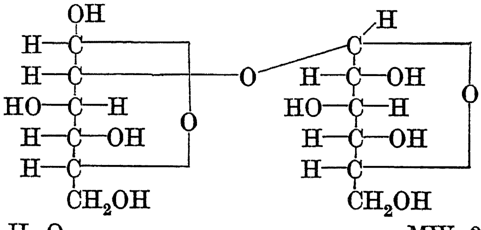
Methiodide: m.p. 234–6°.

Picrolonate: m.p. 226–8° decomp.

Orechoff, *Chem. Zentr.*, 1935, II, 2215.
Orechoff, Proskurnina, Konowalowa, *Ber.*, 1935, **68**, 431.

Sophorine.
See Cytisine.

Sophorose (2-β-d-*Glucosido*-d-*glucose*)



$C_{12}H_{22}O_{11}$ MW, 342

Disaccharide from *Sophora japonica*, Linn. M.p. 195–6°. $[\alpha]_D^{20}$ + 20° in H_2O (final).

α-Octa-acetyl: m.p. 111°. $[\alpha]_D^{17} + 45°$.
β-Octa-acetyl: m.p. 192°. $[\alpha]_D^{20} - 8°$.

Rabaté, *Bull. soc. chim.*, 1940, **7**, 565.
Freudenberg, Knauber, Cramer, *Ber.*, 1951, **84**, 144.

Sorbic Acid (1 : 3-*Hexadienic acid*, 1 : 3-*pentadiene*-1-*carboxylic acid*, 2-*propenylacrylic acid*)

$CH_3·CH:CH·CH:CH·COOH$

$C_6H_8O_2$ MW, 112

Trans: *trans*-.
Needles from EtOH.Aq. M.p. 134·5°. B.p. 228° decomp. Sol. EtOH, Et_2O. Mod. sol. hot H_2O. $k = 1·73 \times 10^{-5}$ at 25°.
Me ester: leaflets. M.p. 5°. B.p. 180°/759 mm. (174°), 70°/20 mm.
Et ester: b.p. 195·5°, 85°/20 mm., 76·5°/12 mm. D_4^{20} 0·9560. n_D^{20} 1·502.
Phenyl ester: b.p. 141°/2 mm.
Chloride: b.p. 78°/15 mm.
Amide: needles from H_2O. M.p. 168°. Sol. H_2O, EtOH.
Nitrile: b.p. 72°/20 mm., 50–60°/12 mm.
Anilide: m.p. 153°.
Morpholide: needles. M.p. 75–6°.
Phenylhydrazide: m.p. 162–3°.
S-*Benzylthiouronium salt*: m.p. 169–70°.

Cis: *trans*-.
Needles from pentane. M.p. 32–5°. B.p. 121–5°/16 mm.
Me ester: b.p. 60°/11 mm. n_D^{22} 1·4948.
Amide: m.p. 113–14°.
S-*Benzylthiouronium salt*: m.p. 137°.

Philippi, *Monatsh.*, 1929, **51**, 278.
Allen, Van Allan, *Organic Syntheses*, 1944, XXIV, 92.
Eisner, Elvidge, Linstead, *J. Chem. Soc.*, 1953, 1372.

Sorbic Aldehyde (1 : 3-*Hexadienal*, *sorbaldehyde*)

$CH_3·CH:CH·CH:CH·CHO$

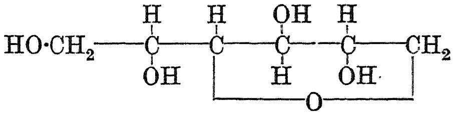
C_6H_8O MW, 96

B.p. 173–4°/754 mm., 76°/30 mm., 64–6°/11 mm. D_4^{22} 0·9087. n_D^{22} 1·5372. Reduces Tollen's rapidly, cold Fehling's slowly.
Oxime: needles from EtOH. M.p. 159·5–160·5° decomp.
Semicarbazone: plates from EtOH. M.p. 206°.
Phenylhydrazone: yellow plates from EtOH. M.p. 101–2°.

Fischer, Wiedemann, *Ann.*, 1934, **513**, 256.
Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, **15**, 261.
Kuhn, Hoffer, *Ber.*, 1930, **63**, 2164; 1931, **64**, 1978.
Baumgarten, Glatzel, *Ber.*, 1926, **59**, 2662.
Langenbeck, Gödde, *Chem. Abstracts*, 1940, **34**, 1970.

Sorbierite.
See Iditol.

Sorbitan (1 : 4-*Anhydro*-D-*sorbitol*, *arlitan*)

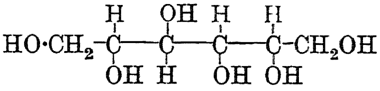


$C_6H_{12}O_5$ MW, 164

Cryst. from isopropyl alcohol. M.p. 115–16°. $[\alpha]_D^{27} - 21·9°$ in H_2O.
Tetra-Me ether: b.p. 245–50°, 170–4°/14 mm. $[\alpha]_D^{22} - 43°$ in EtOH.

Soltzberg, Goepp, Freudenberg, *J. Am. Chem. Soc.*, 1946, **68**, 919.

Sorbitol



$C_6H_{14}O_6$ MW, 182

d-.
Occurs in ripe mountain ash berries, cherries, plums, pears, apples, medlars, fruit of cherry laurel, etc. Needles + ½ or $1H_2O$. M.p. 110–11° (anhyd.), 75° (hydrate), 87–95° from EtOH. Sweet taste. Sol. H_2O. Spar. sol. cold EtOH. $[\alpha]_D^{15} - 1·73°$ in H_2O, $[\alpha]_D^{20} - 2·01°$ in H_2O. HI ⟶ *sec.-n*-hexyl iodide. Sorbose bacterium ⟶ *l*-sorbose.
Triformyl: m.p. 213°.
Hexa-acetyl: cryst. from EtOH. M.p. 99·5° (120°). $[\alpha]_D^{18} + 6·8°$ in Me_2CO.
Dibenzoyl deriv.: m.p. 140°. $[\alpha]_D^{18} + 1·69$–$1·85°$ in Py.
Pentabenzoyl deriv.: m.p. 222°. $[\alpha]_D^{12} + 24·54°$ in Py.
Hexabenzoyl: m.p. 129°. $[\alpha]_D^{19} + 24·3°$ in Py.
Tetra-Me ether: b.p. 125°/0·4 mm. n_D 1·4568. $[\alpha]_D - 6·2°$ in H_2O.
Penta-Et ether: b.p. 185–90°/10 mm.
Triacetone deriv.: m.p. 46–7°. $[\alpha]_D^{15} + 12·7°$ in EtOH.
1 : 3-2 : 4-5 : 6-*Triethylidene deriv.*: needles from petrol. M.p. 94–5°.
Benzylidene deriv.: cryst. M.p. 175°. $[\alpha]_D + 6°$ in EtOH.
Dibenzylidene deriv.: cryst. M.p. 163°. $[\alpha]_D + 29°$ in Me_2CO.
o-*Chlorobenzylidene deriv.*: m.p. 170°.
Tri-o-*chlorobenzylidene deriv.*: m.p. 217°.
2 : 6-*Dichlorobenzylidene deriv.*: m.p. 204·5°.
2-*Nitro*-5-*chlorobenzylidene deriv.*: m.p. 250·5°.
m-*Nitrobenzylidene deriv.*: m.p. 180°.
Di-m-*Nitrobenzylidene deriv.*: m.p. 228·5°.
Tri-o-*nitrobenzylidene deriv.*: m.p. 181° (two forms, m.p. 212–15° and 142–6°).
Tri-m-*nitrobenzylidene deriv.*: m.p. 168°.

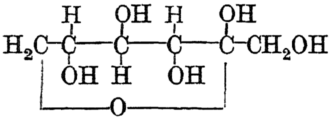
l-.

Needles + $\frac{1}{2}H_2O$. M.p. 77°.

Dibenzylidene deriv.: cryst. M.p. 160°. $[\alpha]_D$ − 28°.

Boussingault, *Ann. chim. phys.*, 1872, **26**, 376.
Meunier, *Compt. rend.*, 1890, **111**, 49.
Vincent, Delachanal, *ibid.*, 51.
Fischer, *Ber.*, 1890, **23**, 3684.
Fischer, Stahel, *Ber.*, 1891, **24**, 2144.
Lobry de Bruyn, v. Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151; 1900, **19**, 7.
Asahina, Shinoda, *J. Pharm. Soc. Japan*, 1930, **50**, 1.
Davis, Slater, Smith, *Biochem. J.*, 1926, **20**, 1155.
Böeseken, Leefers, *Rec. trav. chim.*, 1935, **54**, 861.
Bleyer, Diemair, Lix, *Chem. Abstracts*, 1933, **27**, 4624.
Borisoglebskii, *J. Applied Chem., U.S.S.R.*, 1940, **13**, 571, (*Chem. Abstracts*, 1941, **35**, 1032).
Bourne, Wiggins, *J. Chem. Soc.*, 1948, 1933.

Sorbose



$C_6H_{12}O_6$ MW, 180

d-.

Cryst. M.p. 165°. Sweet taste. Sol. H_2O. Spar. sol. MeOH, EtOH. $[\alpha]_D^{20}$ + 42·9° in H_2O. D^{15} 1·654. Reduces Fehling's. Non-fermentable.

Phenylosazone: m.p. 168° (156°, 160°). Identical with *d*-gulosazone and *d*-idosazone.

β-*Methylglycoside*: cryst. M.p. 119°. $[\alpha]_D$ + 88·5° in H_2O.

l-.

Cryst. M.p. 165° (162°, 159–61°). Sweet taste. Sol. H_2O. Spar. sol. EtOH. $[\alpha]_D^{20}$ − 42·9° in H_2O (− 43·2° in H_2O). D^{15} 1·654. Reduces Fehling's. Heat of comb. C_v 3714·5 Cal. NaHg ⟶ sorbitol + iditol. Non-fermentable.

Phenylosazone: yellow needles. M.p. 168° (164, 156°). Identical with *l*-gulosazone and *l*-idosazone. p-*Bromophenylosazone*: yellow needles. M.p. 181°.

o-*Nitrophenylosazone*: red powder. M.p. 211–12°.

β-*Methylglycoside*: plates from Me_2CO. M.p. 120–2°. $[\alpha]_D^{20}$ − 88·5° in H_2O.

Penta-acetyl: m.p. 96·5–97·5°. $[\alpha]_D$ + 2·9° in $CHCl_3$.

1 : 3 : 4 : 5-*Tetra-Me ether*: b.p. 64°/0·08 mm. $[\alpha]_D^{20}$ − 14·6° in $CHCl_3$.

1 : 3 : 4 : 6-*Tetra-Me ether*: b.p. 64°/0·01 mm. $[\alpha]_D^{20}$ + 29·7° in $CHCl_3$. *Methylglycoside*: b.p. 51°/0·01 mm. $[\alpha]_D^{20}$ − 39·4° in $CHCl_3$.

dl-. β-Acrose.

Laminæ. M.p. 162–3° (154°). D^{17} 1·638 (1·634). Non-fermentable.

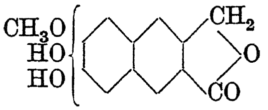
Phenylosazone: laminæ from dil. EtOH. M.p. 169–70° decomp.

Schmitz, *Ber.*, 1913, **46**, 2334.
v. Ekenstein, Blanksma, *Rec. trav. chim.*, 1908, **27**, 1.
Schlubach, Vorwerk, *Ber.*, 1933, **66**, 1251.
Maurer, Schiedt, *Biochem. Z.*, 1934, **271**, 61.
Reichstein, Grussner, *Helv. Chim. Acta.*, 1934, **17**, 318.
Talen, *Rec. trav. chim.*, 1925, **44**, 891.
Böeseken, Leefers, *Rec. trav. chim.*, 1935, **54**, 861.
Fischer, Baer, *Helv. Chim. Acta*, 1936, **19**, 519.
Bernhauer, Görlich, *Biochem. Z.*, 1935, **280**, 375.

Sorbyl Alcohol.

See 2 : 4-Hexadienol-1.

α-Sorigenin



Proposed structure

$C_{13}H_{10}O_5$ MW, 246

Pale yellow needles. M.p. 227–9° decomp. Monobasic.

Di-Me ether: m.p. 183·5–184·5°.

Diacetyl: m.p. 255–9°.

Nikuni, *Chem. Abstracts*, 1940, **34**, 3259; 1939, **33**, 6301; 1938, **32**, 7469.

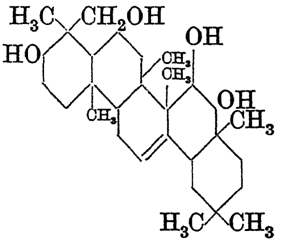
α-Sorinin

$C_{24}H_{28}O_{14}$ MW, 540

Isolated from bark of *Rhamnus japonica*, Max. Needles from MeOH.Aq. M.p. 159°. Boiling H_2O ⟶ α-sorigenin + primeverose.

Nikuni, *Chem. Abstracts*, 1939, **33**, 6301; 1938, **32**, 7469.

Soya-sapogenol A



Suggested structure

$C_{30}H_{50}O_4$ MW, 474

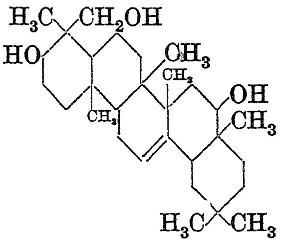
Triterpene alcohol from soya bean saponin. Leaflets from MeOH. M.p. 308–12°. $[\alpha]_D^{21}$ + 102·3° in $CHCl_3$.

Tetra-acetyl: m.p. 232°. $[\alpha]_D^{15}$ + 86·1° in $CHCl_3$.

Tribenzoyl: m.p. 255°.

Tsuda, Kitewaga, *Ber.*, 1938, **71**, 790; 1937, **70**, 2083.
Miyasaka, *J. Pharm. Soc. Japan*, 1937, **57**, 464.
Meyer, Jeger, Ruzicka, *Helv. Chim. Acta*, 1950, **33**, 672.

Soya-sapogenol B



Suggested structure

$C_{30}H_{50}O_3$ MW, 458

Triterpene alcohol from soya bean saponin. Needles from MeOH. M.p. 258–9°. $[\alpha]_D^{30.5}$ + 92·4° in $CHCl_3$.

Triformyl: prisms from Me_2CO–Et_2O. M.p. 218°.

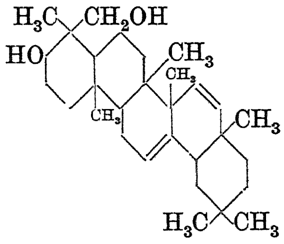
Triacetyl: plates from MeOH. M.p. 175–6°. $[\alpha]_D^{16}$ + 83° in $CHCl_3$. *Dibromide*: needles from Me_2CO–MeOH. M.p. 225–7°.

Tribenzoyl: m.p. 133°.

Tri-p-*bromobenzoyl*: cryst. from Me_2CO. M.p. 255–7°.

Tsuda, Kitewaga, *Ber.*, 1938, **71**, 790; 1937, **70**, 2083.
Miyasaka, *J. Pharm. Soc. Japan*, 1937, **57**, 464.
Meyer, Jeger, Ruzicka, *Helv. Chim. Acta*, 1950, **33**, 687.

Soya-sapogenol C



Suggested structure

$C_{30}H_{48}O_2$ MW, 440

Triterpene alcohol from soya bean saponin. Needles from MeOH. M.p. 238–9°. $[\alpha]_D^{31}$ + 70·7° in $CHCl_3$.

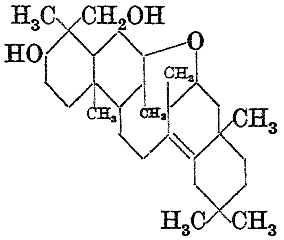
Diformyl: prisms from Me_2CO–Et_2O. M.p. 265°.

Diacetyl: plates from MeOH–EtOH. M.p. 198°. $[\alpha]_D$ + 59°. *Dibromide*: needles from Me_2CO–Et_2O. M.p. 225–7°.

Dibenzoyl: cryst. from Me_2CO–MeOH. M.p. 188°.

Tsuda, Kitewaga, *Ber.*, 1938, **71**, 790; 1937, **70**, 2083.
Miyasaka, *J. Pharm. Soc. Japan*, 1937, **57**, 464.
Meyer, Jeger, Ruzicka, *Helv. Chim. Acta*, 1950, **33**, 672.

Soya-sapogenol D



Suggested structure

$C_{30}H_{50}O_3$ MW, 458

Triterpene alcohol from soya bean saponin. Prisms from Me_2CO–MeOH. M.p. 298–9°. $[\alpha]_D^{31}$ − 60·8° in $CHCl_3$.

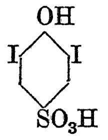
Diformyl: needles from Me_2CO–Et_2O. M.p. 231°.

Diacetyl: prisms from MeOH. M.p. 192°. $[\alpha]_D$ − 45°.

Dibenzoyl: needles from Me_2CO. M.p. 240°.

Tsuda, Kitewaga, *Ber.*, 1938, **71**, 790; 1937, **70**, 2083.
Miyasaka, *J. Pharm. Soc. Japan*, 1937, **57**, 464.
Meyer, Jeger, Ruzicka, *Helv. Chim. Acta*, 1950, **33**, 687.

Sozoiodolic Acid (2 : 6-*Di-iodophenol*-p-*sulphonic acid*)



$C_6H_4O_4I_2S$ MW, 426

Prisms + $3H_2O$. M.p. 120–120·5°. Sol. H_2O. CrO_3 + H_2SO_4 ⟶ 2 : 6-di-iodo-*p*-benzoquinone.

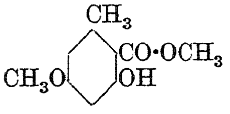
Et ether: $C_8H_8O_4I_2S$. MW, 454. Cryst. + $2H_2O$. M.p. 108°. Sol. H_2O.

d-*Arginine salt*: decomp. at 213–14°.
Cadaverine salt: decomp. at 242°.
Choline salt: decomp. at 180°.
Creatinine salt: decomp. at 229–31°.
Glucosamine salt: decomp. at 181–2°.
Guanidine salt: decomp. at 247–9°.
Histamine salt: decomp. at 241°.
l-*Histidine salt*: decomp. at 207–8°.
Lysine salt: decomp. at 234–5°.
Putrescine salt: decomp. at 250°.

Urea salt : decomp. at 208°.

Tromsdorff, D.R.P., 45,226.
Rupp, Herrmann, *Arch. Pharm.*, 1916, **254**, 509.
Ackermann, *Z. physiol. Chem.*, 1934, **225**, 46.

Sparassol (*Methyl everninate, 3-hydroxy-5-methoxy-*o*-toluic acid methyl ester*)



$C_{10}H_{12}O_4$ MW, 196

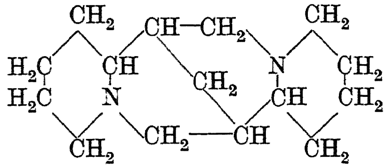
Occurs in *Evernia prunastri*, Ach., *Sparassis ramosa*, Schäff., and *Rhododendron japonicum*, C. J. Schneider. M.p. 67–8°. $FeCl_3 \longrightarrow$ violet col. in EtOH.

Acetyl : m.p. 41–2°.

3-*Me ether* : $C_{11}H_{14}O_4$. MW, 210. M.p. 41–2°.

Kinoshita, *Chem. Abstracts*, 1931, **25**, 1552.
Wedekind, Fleischer, *Ber.*, 1924, **57**, 1121.
Späth, Jesckki, *ibid.*, 471.
Pfau, *ibid.*, 468.
Falck, *Ber.*, 1923, **56**, 2555.
Koller, Hamburg, *Monatsh.*, 1935, **65**, 375.

Sparteine (*Lupinidine, hexahydrodeoxy-anagyrine, pachycarpine*)



$C_{15}H_{26}N_2$ MW, 234

d-.

Occurs in seeds of *Anagyris fœtida*, Linn, *Sophora pachycarpa*, Schrenk, *Thermopsis lanceolata*, R.Br. B.p. 173–4°/8 mm., 138°/2 mm., 133–5°/1 mm. Sol. EtOH, Et_2O, $CHCl_3$. Insol. H_2O, C_6H_6, ligroin. D_4^{20} 1·027. n_D^{20} 1·5312. $[\alpha]_D^{20}$ + 16·1° (+ 5·54°). Volatile in steam. Resinifies in air.

B,2HCl : m.p. 255–7°.

B,HI : m.p. 232–3°.

$B,HClO_4$: plates from EtOH. M.p. 171–2°.

B,H_2PtCl_6 : m.p. 240–1° decomp.

Chloroaurate : m.p. 192–3°.

Dipicrate : yellow needles from EtOH-Me_2CO. M.p. 205–6°.

Methiodide : m.p. 236–8°. $[\alpha]_D$ + 24·5° in H_2O.

l-.

B.p. 130–5°/1 mm. $[\alpha]_D$ — 11·3° in EtOH.

B,HI : prisms from H_2O. M.p. 231°.

Dipicrate : m.p. 205–6°.

dl-.

Monopicrate : m.p. 134–5°.

Dipicrate : m.p. 205–6°.

Mills, *Ann.*, 1863, **125**, 71.
Clemo, Leitch, Raper, *Ber.*, 1931, **64**, 1520.
Clemo, Raper, *J. Chem. Soc.*, 1933, 644.
Clemo, Raper, Tenniswood, *J. Chem. Soc.*, 1931, 429.
Orechoff, Proskurnina, *Ber.*, 1935, **68**, 1807.
Leonard, Beyler, *J. Am. Chem. Soc.*, 1948, **70**, 2298.

Spartioidine

$C_{18}H_{23}O_5N$ MW, 333

Alkaloid from *Senecio spartioides*. Prisms from MeOH. M.p. 178°.

Methiodide : plates from $CHCl_3$. M.p. 239° decomp.

Manske, *Can. J. Research.*, 1939, **17**, 1.

Spathulatine

$C_{33}H_{64}O_5N_4$ MW, 596

Alkaloid from *Lupinus sericeus* and *L. spathulatus*. Cryst. from Me_2CO. M.p. 233–4° corr. Sol. H_2O, EtOH, C_6H_6, $CHCl_3$. Spar. sol. Et_2O, pet. ether. Sols. are slightly lævorotatory. Sensitive to strong min. acids.

B,3KI : prisms from H_2O. M.p. 260–1°.

Picrate : yellow needles. M.p. 182–4°.

Methiodide : cryst. M.p. 250–2°.

Couch, *J. Am. Chem. Soc.*, 1940, **62**, 554; 1924, **46**, 2507.

Spermidine (*ω-Aminopropyl-ω-aminobutyl-amine*)

$H_2N{\cdot}CH_2{\cdot}[CH_2]_2{\cdot}NH{\cdot}[CH_2]_3{\cdot}CH_2NH_2$

$C_7H_{19}N_3$ MW, 145

Occurs in ox pancreas as phosphate. B.p. 128–30°/14 mm.

$B_2,(H_3PO_4)_3,6H_2O$: plates from EtOH.Aq. M.p. 207–8°. Sol. H_2O.

$B,3HAuCl_4$: needles from 1% HCl. M.p. 222°.

*Tri-*m*-nitrobenzoyl* : m.p. anhyd. 148–50°.

Tripicrate : yellow needles from H_2O. M.p. 211°.

Dudley, Rosenheim, Starling, *Biochem. J.*, 1927, **21**, 97.
v. Braun, Pinkernelle, *Ber.*, 1937, **70**, 1233.

Spermine (*Gerontine, musculamine, neuridine, di-[ω-aminopropyl]-tetramethylenediamine*)

$H_2N{\cdot}CH_2{\cdot}[CH_2]_2{\cdot}NH{\cdot}[CH_2]_4{\cdot}NH{\cdot}[CH_2]_2{\cdot}CH_2NH_2$

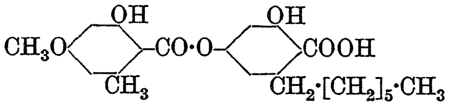
$C_{10}H_{26}N_4$ MW, 202

Occurs as phosphate in semen, ox pancreas, yeast, etc. Deliquescent cryst. Absorbs CO_2 from the air.

Phosphate: m.p. 230–4°. Sol. hot H_2O. Insol. EtOH, Et_2O.
Tetrabenzoyl: needles from Me_2CO.Aq. M.p. 155°.
B,4HCl: needles. M.p. 310° decomp.
B,4HAuCl$_4$: golden leaflets from 5% HCl. M.p. 226° decomp.
B,2H$_2$PtCl$_6$: m.p. 242–5°.
Tetrapicrate: yellow needles from H_2O. M.p. 248–50°.
Picrolonate: pale yellow needles from H_2O. M.p. 288–9° decomp.

Wrede, Fanselow, Strack, *Z. physiol. Chem.*, 1927, **163**, 219.
Dudley, Rosenheim, Rosenheim, *Biochem. J.*, 1924, **18**, 1263.
Wrede, Strack, Hettche, *Z. physiol. Chem.*, 1928, **173**, 61.
Dudley, Rosenheim, Starling, *Biochem. J.*, 1926, **20**, 1082.

Sphærophorin

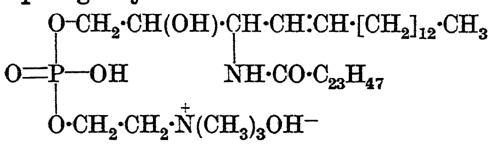


$C_{23}H_{28}O_7$ MW, 416

Depside from the lichen *Sphærophorus melanocarpus*. Plates or needles from C_6H_6. M.p. 140°. Alc. KOH ⟶ everninic acid + 5-heptylresorcinol, m.p. 57–8°.
Diacetyl: needles from 80% EtOH. M.p. 133–4°.
Di-Me ether–Me ester: prisms from 80% EtOH. M.p. 85–6°.

Asahina, Hashimoto, *Ber.*, 1934, **67**, 416.

Sphingomyelin



$C_{47}H_{97}O_7N_2P$ MW, 832

A phospholipid. Crystallises as thin plates. Insol. Et_2O.

Levene, *J. Biol. Chem.*, 1913, **15**, 153; 1914, **18**, 453, 1916, **24**, 69.

Sphingosine

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH{=}CH{-}CH(NH_2){-}CH(OH){-}CH_2OH$

$C_{18}H_{37}O_2N$ MW, 299

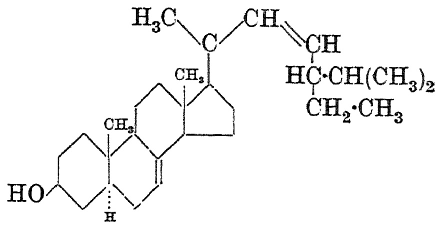
Needles from Et_2O. M.p. 79–81°.
B$_2$,H$_2$SO$_4$: m.p. 233–4° decomp.
Triacetyl deriv.: m.p. 102–3°. $[\alpha]_D^{19}$ − 24·09°.
Tribenzoyl deriv.: cryst. from EtOH. M.p. 121·5–123·5°. $[\alpha]_D^{27}$ −11·2°.
Lignoceryl deriv.: m.p. 90–90·5°.
Picrolonate: m.p. 87–9°.

Me ether: *B,HCl*, plates from Me_2CO–EtOH. M.p. 141°.
Di-Me ether: *B,HCl*, plates from Me_2CO–EtOH. M.p. 139° (133–4°).
Di-Et ether: *B,HCl*, plates from Me_2CO. M.p. 113–15°.
Galactoside: psychosine. Cryst. *Sulphate*: m.p. 225° decomp. $[\alpha]_D$ − 16·6°.

Levene, Jacobs, *J. Biol. Chem.*, 1912, **11**, 547.
Klenk, Diebold, *Z. physiol. Chem.*, 1931, **198**, 25.

Spinacene.
See Squalene.

Spinasterol



$C_{29}H_{48}O$ MW, 412

α-.
Occurs in spinach and senega root. Plates from EtOH. M.p. 172–5° (169–70°). $[\alpha]_D^{17}$ − 3·7° in $CHCl_3$.
Acetyl: plates from AcOH. M.p. 187° (183–5°). $[\alpha]_D^{23}$ − 4·7° in $CHCl_3$.
Propionyl: plates from AcOEt. M.p. 152–3°. $[\alpha]_D^{23}$ − 5·0° in $CHCl_3$.
Benzoyl: plates from AcOEt. M.p. 201–2°. $[\alpha]_D^{23}$ + 2·25° in $CHCl_3$.
p-*Nitrobenzoyl*: needles from AcOEt. M.p. 217–18°. $[\alpha]_D^{23}$ + 4·5° in $CHCl_3$.
Phenylurethane: plates from AcOEt. M.p. 177° (173–4°). $[\alpha]_D^{23}$ − 2·25° in $CHCl_3$.

β-.
Occurs in spinach. Cryst. from EtOH. M.p. 145–8°. $[\alpha]_D^{20}$ + 5·9°, $[\alpha]_{5461}$ + 7·65°.
Acetyl: cryst. from AcOH. M.p. 150–4°. $[\alpha]_D$ + 5·1°.
Benzoyl: m.p. 181–3°. $[\alpha]_D^{19}$ +7·5°.

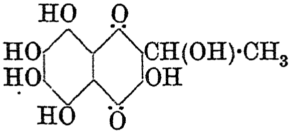
γ-.
Occurs in spinach. M.p. 159–60°.
Acetyl: m.p. 139·5–140°. $[\alpha]_{5461}$ − 14·1° in $CHCl_3$.
Benzoyl: m.p. 118·5–119°. $[\alpha]_{5461}$ − 10·3° in $CHCl_3$.
p-*Nitrobenzoyl*: m.p. 200°. $[\alpha]_{5461}$ − 8·9° in $CHCl_3$.
Phenylurethane: m.p. 144–5°. $[\alpha]_{5461}$ − 15·9° in $CHCl_3$.

δ-.
Occurs in alfalfa seed oil. M.p. 143–4°. $[\alpha]_D$ + 6·2°.
Acetyl: m.p. 132–133·5°. $[\alpha]_D$ + 1°.

Benzoyl: m.p. 165–8°. $[\alpha]_D^{20} + 11{\cdot}1°$.

Simpson, *J. Chem. Soc.*, 1937, 730.
Heyl, Larsen, *J. Am. Chem. Soc.*, 1934, **56**, 942, 2664; *Chem. Zentr.*, 1933, II, 2019.
Hart, Heyl, *J. Biol. Chem.*, 1932, **95**, 311.
King, Ball, *J. Am. Chem. Soc.*, 1942, **64**, 2488.
Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 282.

Spinochrome A



$C_{12}H_{10}O_8$ MW, 282

Pigment of the sea-urchin, North Atlantic *Paracentrotus lividus*. Violet prisms from EtOH. M.p. 185°.

Glaser, Lederer, *Compt. rend.*, 1939, **208**, 1939.
Goodwin, Srisukh, *Biochem. J.*, 1950, **47**, 69.
Goodwin, Lederer, Musajo, *Experientia*, 1951, **7**, 375.

Spinochrome P

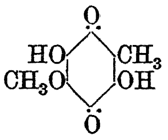
$C_{12}H_{10}O_7$ MW, 266

Pigment of the sea-urchin, Mediterranean *Paracentrotus lividus*. Deep garnet-red platelets from dioxan-H_2O. M.p. 188°. k (first) = $3{\cdot}9 \times 10^{-9}$; (second) = $0{\cdot}6 \times 10^{-11}$. Forms insol. mono-Na, di-Na, Ca salts. Reductive acetylation ⟶ hepta-acetyl dihydro-spinochrome-P, m.p. 178°.

Tri-Me ether: m.p. 165–6°.

Musajo, Minchilli, *Gazz. chim. ital.*, 1940, **70**, 287.
Goodwin, Srisukh, *Biochem. J.*, 1950, **47**, 69.
Goodwin, Lederer, Musajo, *Experientia*, 1951, **7**, 375.

Spinulosin (3 : 6-*Dihydroxy*-5-*methoxytoluquinone*)



$C_8H_8O_5$ MW, 184

Metabolic product of *Penicillium spinulosum*, Thom. Sublimes at 120°/1 mm. in black metallic plates, m.p. 202–3°.

Di-Me ether: $C_{10}H_{12}O_5$. MW, 212. Reddish orange needles from EtOH. M.p. 80°.

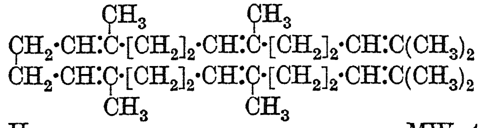
Diacetyl: needles from EtOH. M.p. 139·5°.

Anslow, Raistrick, *Biochem. J.*, 1938, **32**, 803; *J. Chem. Soc.*, 1939, 1446.
Bracken, Raistrick, *Biochem. J.*, 1947, **41**, 569.

Spirozid.

See under 2-Aminophenol-4-arsonic Acid.

Squalene (*Spinacene*)



$C_{30}H_{50}$ MW, 410

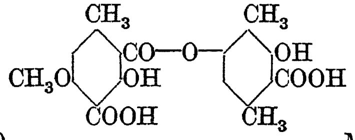
Occurs in oil of *Centrophorus granulosus*, Müller & Henle, *Scymnorhinus lichio*, *Etmopterus spinan*, *Lepidorhinus squamosus*. F.p. below −20°. B.p. 284–5°/25 mm., 280°/17 mm., 260–2°/9 mm., 240–2°/4 mm., 105°/0·17 mm. D_{20}^{20} 0·8592. n_D^{20} 1·4990.

HCl add. comp.: (i) m.p. 114°. (ii) M.p. 143–4°.

HBr add. comp.: (i) m.p. 116–18°. (ii) M.p. 136–8°.

Challenger, *Industrial Chemist*, 1928, **4**, 315 (*Bibl.*).
Karrer, Helfenstein, *Helv. Chim. Acta*, 1931, **14**, 78.
Heilbron, Kamm, Owens, *J. Chem. Soc.*, 1926, 1630.
Schmitt, *Ann.*, 1941, **547**, 115.

Squamatic Acid



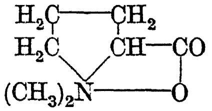
$C_{19}H_{18}O_9$ MW, 390

Occurs in the lichen *Cladonia bellidiflora*. Prisms from AcOH. M.p. 219° (228°, 215°). Sol. AcOH, AcOEt. Spar. sol. EtOH, Et_2O, $CHCl_3$.

Di-Me ester: $C_{21}H_{22}O_9$. MW, 418. M.p. 178°. *Di-Me ether*: $C_{23}H_{26}O_9$. MW, 446. Leaflets from MeOH. M.p. 135°.

Asahina, Tanase, *Ber.*, 1937, **70**, 62.
Asahina, Sakurai, *ibid.*, 64.

Stachydrine (*Hygric acid methylbetaine*, N-*methylproline methylbetaine*)



$C_7H_{13}O_2N$ MW, 143

Occurs in *Stachys tuberifera*, Naudin, and in fruit of *Citrus grandis*, Hassk, etc. Cryst. + $1H_2O$. M.p. anhyd. 235° decomp. Sol. H_2O, EtOH. Insol. Et_2O, $CHCl_3$. Decomp. in air.

B,HCl: cryst. from EtOH. M.p. 235° (211–15°).

B_2,H_2PtCl_6: plates from 50% EtOH. Decomp. at 200°.

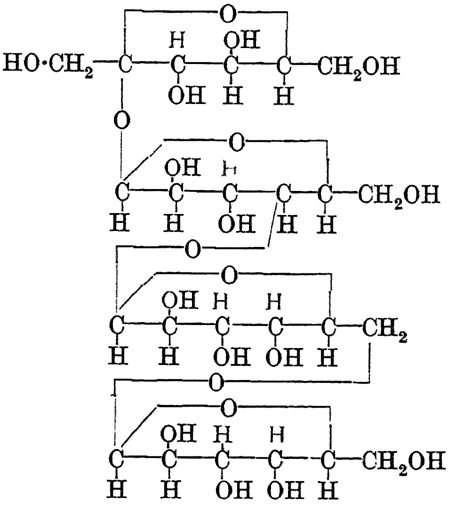
B, $HAuCl_4$: m.p. 225–6°.

B,$(COOH)_2$: needles. M.p. 105–7°.

Picrate: needles. M.p. 195–6°.

Schulze, Trier, *Z. physiol. Chem.*, 1910, **67**, 59; *Ber.*, 1909, **42**, 4654.
Steenbock, *J. Biol. Chem.*, 1918, **35**, 1.
Kuhn, Brydówna, *Ber.*, 1937, **70**, 1333.

Stachyose



$C_{24}H_{42}O_{21}$ MW, 666

Occurs in ash manna, seeds of *Leguminosœ*, roots and rhizomes of *Labiates*, e.g., *Stachys tuberifera.* Plates + $4H_2O$ (or $1\frac{1}{2}H_2O$). Loses H_2O at 115° in vacuum. M.p. anhyd. 167–70°. Sol. H_2O. Insol. EtOH, Et_2O. Sweet taste. $[\alpha]_D^{15}$ + 133·9° in H_2O, + 148·4° (anhyd.) in H_2O. Does not reduce Fehling's. Does not form an osazone. Partly fermentable. Hyd. by dil. acids ⟶ glucose (1 mol.) + galactose (2 mols.) + fructose (1 mol.). Heat of comb. C_v = 3808 Cal. Forms compounds with BaO and SrO.

Tetradeca-acetyl: m.p. 95–6°. $[\alpha]_D^{22\cdot5}$ + 120·2° in EtOH, $[\alpha]_D^{26}$ + 120·5° in AcOH.

*Tetradeca-*p*-nitrobenzoyl*: m.p. 166°.

Tetradeca-Me ether: syrup. $[\alpha]_D^{24\cdot5}$ + 133·3° in C_6H_6, + 139·2° in $CHCl_3$, + 129·9° in Me_2CO, + 135·2° in EtOH, + 137·1° in MeOH, + 120·3° in Et_2O.

Tanret, *Compt. rend.*, 1912, **155**, 1526.
Neuberg, Lachmann, *Biochem. Z.*, 1910, **24**, 171.
Onuki, *Chem. Abstracts*, 1932, **26**, 4308; 1933, **27**, 2138.

Starch (*Amylum*)

$(C_6H_{10}O_5)_n$ MW, $(162)_n$

The main reserve carbohydrate in the vegetable kingdom. Consists of two carbohydrates (polyglucoses), amylose and amylopectin. White granular powder. Insol. cold H_2O, EtOH, Et_2O. When heated with water swells and gives an opalescent sol. Hyd. by acids ⟶ dextrins ⟶ maltose ⟶ glucose.

Amylose is a linear polymer composed of glucose units joined by 1 : 4-α-linkages. Gives deep blue col. with I.

Amylopectin is a branched polymer composed of short chains of 1 : 4-α-linked glucose units, the chains being joined by cross-linkages, some, if not all, of which are of the 1 : 6-type.

Starch and its Derivatives, Edited by Radley.
The Chemistry and Industry of Starch, Edited by Kerr.
Bourne, *Chemistry and Industry*, 1951, 1047 (*Review*).

Stearaldehyde.
See Stearic Aldehyde.

Stearamide (*Stearic acid amide*)

$CH_3\cdot[CH_2]_{16}\cdot CONH_2$

$C_{18}H_{37}ON$ MW, 283

Cryst. from hot EtOH. M.p. 108·5–109°. B.p. 250°/12 mm. (168–9° *in vacuo*). Sol. Et_2O, $CHCl_3$, hot EtOH. Insol. H_2O.

Aschan, *Ber.*, 1898, **31**, 2349.

Stearanilide (*Stearic acid anilide*)

$CH_3\cdot[CH_2]_{16}\cdot CO\cdot NH\cdot C_6H_5$

$C_{24}H_{41}ON$ MW, 359

Needles from EtOH. M.p. 94° (88°). B.p. 153·5°/10 mm. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, MeOH, Me_2CO, hot AcOH. Spar. sol. pet. ether. Insol. H_2O.

De' Conno, *Gazz. chim. ital.*, 1917, **47**, i, 98.
Nill, U.S.P., 1,659,150, (*Chem Abstracts*, 1928, **22**, 1367).

Stearic Acid (*Octadecylic acid*)

$CH_3\cdot[CH_2]_{16}\cdot COOH$

$C_{18}H_{36}O_2$ MW, 284

Occurs in moulds, in jalap resin, in fruits of *Citrullus colcynthis*, Schrad., and as esters (glycerides) in fats. Leaflets from EtOH. F.p. 69·41°. M.p. 71·5–72° (69·6°, 69·5°). B.p. 360° decomp., 291°/100 mm., 238°/17 mm., 232°/15 mm., 158–60°/0·25 mm. Mod. sol. hot EtOH. Spar. sol. C_6H_6, CS_2. D_4^{20} 0·9408. n_D^{70} 1·4335. Heat of comb. 2711·8 Cal.

Li salt: m.p. 220–221·5°.

Cu salt: m.p. 115–20°.

Ag salt: m.p. 205°.

Mg salt: m.p. 132°.

Pb salt: m.p. 115·6–115·8° (113–14°).

Co salt: m.p. 73–5°.

Ni salt: m.p. 80–6°.

Me ester: $C_{19}H_{38}O_2$. MW, 298. M.p. 38·5–39·5° (39·7–40°). B.p. 442–3°/747 mm., 214–15°/15 mm.
Et ester: $C_{20}H_{40}O_2$. MW, 312. F.p. 30·92°. (i) M.p. 33·4°. (ii) M.p. 30·9°. B.p. 213–15°/15 mm., 199°/10 mm., 152°/0·18 mm.
2-*Chloroethyl ester*: $C_{20}H_{39}O_2Cl$. MW, 346·5. Leaflets from EtOH. M.p. 49·5°.
2-*Iodoethyl ester*: $C_{20}H_{39}O_2I$. MW, 438. Leaflets from EtOH. M.p. 59·5°.
Propyl ester: $C_{21}H_{42}O_2$. MW, 326. Prisms from pet. ether. M.p. 28·6°.
Isobutyl ester: $C_{22}H_{44}O_2$. MW. 340. M.p. 25°. B.p. 199°/5 mm. D_4^{29} 0·8498. $[\alpha]_D^{20}$ + 9·39° in EtOH.
Isoamyl ester: $C_{23}H_{46}O_2$. MW, 354. M.p. 25·5° (22°).
Hexadecyl ester: $C_{34}H_{68}O_2$. MW, 508. M.p. 55–60°.
Heptadecyl ester: $C_{35}H_{70}O_2$. MW, 522. M.p. 64·7°.
Ethylene glycol di-ester: $C_{38}H_{74}O_4$. MW, 594. M.p. 79° (76°).
Propylene glycol di-ester: $C_{39}H_{76}O_4$. MW, 608. M.p. 40°.
Glycerol ester: *see* Monostearin, Distearin, Tristearin.
Phenyl ester: $C_{24}H_{40}O_2$. MW, 360. M.p. 52°. B.p. 267°/15 mm.
o-*Nitrophenyl ester*: $C_{24}H_{39}O_4N$. MW, 405. M p. 60–1°.
p-*Tolyl ester*: $C_{25}H_{42}O_2$. MW, 374. M.p. 54°. B.p. 276°/15 mm.
Benzyl ester: $C_{25}H_{42}O_2$. MW, 374. M.p. 28°.
l-*Menthyl ester*: $C_{28}H_{54}O_2$. MW, 422. Plates from EtOH. M.p. 39°. $[\alpha]_D^{20}$ − 36·71° in $CHCl_3$.
Chloride: $C_{18}H_{35}OCl$. MW, 302·5. M.p. 23°. B.p. 202–3°/6 mm. (215°/5 mm.).
Amide: *see* Stearamide.
Anhydride: $C_{36}H_{70}O_3$. MW, 550. M.p. 72°.
Nitrile: *see* Stearonitrile.
Anilide: *see* Stearanilide.
o-*Chloroanilide*: m.p. 67–68·5°.
p-*Chloroanilide*: m.p. 101–2°.
p-*Bromoanilide*: m.p. 114°.
2 : 4 : 6-*Tribromoanilide*: m.p. 126°.
p-*Nitroanilide*: m.p. 94·5–95·5°.
o-*Toluidide*: m.p. 97°.
p-*Toluidide*: m.p. 102° (95·5°). B.p. 161·5°/10 mm.
1-*Naphthalide*: m.p. 110·8°. B.p. 205°/10 mm.
2-*Naphthalide*: m.p. 112°. B.p. 239°/10 mm.

Hell, Sadomski, *Ber.*, 1891, **24**, 2388.
Heintz, *J. prakt. Chem.*, 1855, **66**, 22.
Veselý, *Chimie et industrie*, 1929, **22**, 881 (*Bibl.*).
Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 965.

Stearic Aldehyde (*Stearaldehyde*)

$CH_3{\cdot}[CH_2]_{16}{\cdot}CHO$

$C_{18}H_{36}O$ MW, 268

M.p. 55° (38°). Polymerizes rapidly.
Oxime: needles. M.p. 89°.
Semicarbazone: needles. M.p. 108–9°.
Thiosemicarbazone: needles from EtOH. M.p. 111°.
p-*Nitrophenylhydrazone*: yellow needles from MeOH. M.p. 101°.

Stephen, *J. Chem. Soc.*, 1925, 1876.
Feulgen, Behrens, *Z. physiol. Chem.*, 1928, **177**, 221.

Note.—Compounds described in earlier literature are polymers.

Stearin.
See Monostearin, Distearin *and* Tristearin.

Stearolactone.
See under 3-Hydroxystearic Acid.

Stearolic Acid

$CH_3{\cdot}[CH_2]_7C{:}C{\cdot}[CH_2]_7{\cdot}COOH$

$C_{18}H_{32}O_2$ MW, 280

Needles from EtOH.Aq. M.p. 47–8°. Sol. Et_2O, hot EtOH. Insol. H_2O. Heat of comb. C_v 2624·7 Cal.
Tetrabromide: *see* 8 : 8 : 9 : 9-Tetrabromostearic Acid.
Et ester: b.p. 195–7°/3 mm.
1-*Glycerol ester*: $C_{21}H_{38}O_4$. MW, 354. Leaflets from EtOH. M.p. 40·5°.
1 : 2-*Glycerol di-ester*: $C_{39}H_{68}O_5$. MW, 616. M.p. 40°.
1 : 3-*Glycerol di-ester*: m.p. 38·5°.
Glycerol tri-ester: $C_{57}H_{98}O_6$. MW, 878. M.p. 29°.
Hydrazide: m.p. 75–75·5°.

Hoffmann-La Roche, D.R.P., 243,582, (*Chem. Zentr.*, 1912, I, 695).
Kino, *Chem. Abstracts*, 1930, **24**, 1998; *Chem. Zentr.*, 1936, I, 2531.
Adkins, Burks, *Organic Syntheses*, 1947, XXVII, 76.
See also Posternak, *Compt. rend.*, 1916, **162**, 944.

Stearone (*Di*-n-*heptadecyl ketone*, 18-*pentatricontanone*)

$CH_3{\cdot}[CH_2]_{16}{\cdot}CO{\cdot}[CH_2]_{16}{\cdot}CH_3$

$C_{35}H_{70}O$ MW, 506

Leaflets from ligroin. M.p. 88·4°. Spar. sol. hot EtOH, hot Et_2O. Insol. H_2O. D_4^{95} 0·7932.
Oxime: m.p. 62–3°.

Easterfield, Taylor, *J. Chem. Soc.*, 1911, **99**, 2300.
Grün, D.R.P., 296,677, (*Chem. Zentr.*, 1917, I, 611).
Kino, Kato, *Chem. Abstracts*, 1940, **34**, 6220.

Stearonitrile (*Stearic acid nitrile, heptadecyl cyanide*)

$CH_3 \cdot [CH_2]_{16} \cdot CN$

$C_{18}H_{35}N$ MW, 265

M.p. 42·5–43·5°. B.p. 214°/13 mm. (128° *in vacuo*). D_4^{41} 0·8178. n_D^{45} 1·4389.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.
van Epps, Reid, *J. Am. Chem. Soc.*, 1916, **38**, 2125.
Kaplan, U.S.P. 2,444,828, (*Chem. Abstracts*, 1948, **42**, 7315).

Stearophenone (*Heptadecyl phenyl ketone*)

$CH_3 \cdot [CH_2]_{15} \cdot CH_2 \cdot CO \cdot C_6H_5$

$C_{24}H_{40}O$ MW, 344

Plates. M.p. 64°. Sol. $CHCl_3$, Et_2O, hot EtOH.
Oxime: cryst. from EtOH. M.p. 53°.
Phenylhydrazone: needles from EtOH. M.p. 54°.

Claus, Häfeln, *J. prakt. Chem.*, 1896, **54**, 399.
Ryan, Nolan, *Chem. Zentr.*, 1913, II, 2050.

Stearoxylic Acid (8 : 9-*Diketostearic acid*)

$CH_3 \cdot [CH_2]_7 \cdot CO \cdot CO \cdot [CH_2]_7 \cdot COOH$

$C_{18}H_{32}O_4$ MW, 312

Yellow plates. M.p. 86° (83–4°). Sol. Et_2O, C_6H_6, hot EtOH. Spar. sol. ligroin.
Monoxime: m.p. 76–81°.
Dioxime: m.p. 153–4°.
2 : 4-*Dinitrophenylosazone*: orange cryst. M.p. 146·5°.

Spieckermann, *Ber.*, 1895, **28**, 276.
Böeseken, *Rec. trav. chim.*, 1911, **30**, 146.

Stearyl Alcohol.
See Octadecyl Alcohol.

Stearylamine.
See Octadecylamine.

Stemonidine

$C_{19}H_{31}O_5N$ MW, 353

Alkaloid isolated from roots of *Stemona japonica*. M.p. 116°. $[\alpha]_D^{12}$ − 7·65°. Tertiary base.
B,HCl: decomp. at 260°.
Methiodide: decomp. at 248°.

Suzuki, *J. Pharm. Soc. Japan*, 1929, **49**, 457; 1939, **59**, 184.

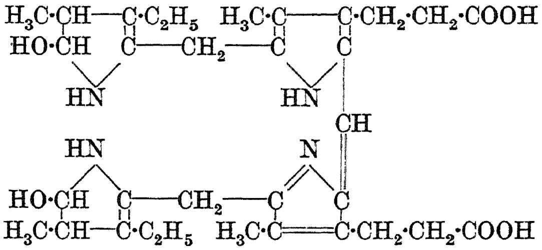
Stemonine

$C_{22}H_{33}O_4N$ MW, 375

White needles. M.p. 160°. $[\alpha]_D$ + 76·5°. Sol. EtOH, Me_2CO, Et_2O, C_6H_6, $CHCl_3$.
Au salt: m.p. 194° decomp.
Me stemonine methiodide: m.p. 265° decomp.

Suzuki, *Chem. Abstracts*, 1931, **25**, 4551.
Lobstein, Grumbach, *Chem. Abstracts*, 1932, **26**, 2012.
Kondo, Satomi, *J. Pharm. Soc. Japan*, 1947, **67**, 182.

Stercobilin

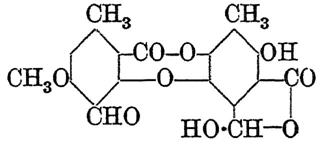


$C_{33}H_{46}O_6N_4$ MW, 594

M.p. 238°. $[\alpha]_{6560}^{20}$ − 824° in AcOH. H_2SO_4 ⟶ glaucobilin.
B,HCl: $[\alpha]_{6560}^{20}$ − 2000° in $CHCl_3$.

Fischer, Halbach, *Z. physiol. Chem.*, 1936, **238**, 59.

Stictic Acid (*Scopuloric acid*)

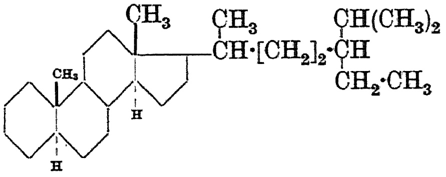


$C_{19}H_{14}O_9$ MW, 386

Occurs in lichen *Ramalina scopulorum*. Needles from Me_2CO. M.p. 270°. $FeCl_3$ ⟶ purple ⟶ brown col.
Diacetyl deriv.: m.p. 235–6°.
Tetra-acetyl deriv.: m.p. 226–7°.
Di-Me ether: $C_{21}H_{18}O_9$. MW, 414. (i) M.p. 174°. (ii) M.p. 242–3°.

Curd, Robertson, *J. Chem. Soc.*, 1935, 1379.
Asahina *et al.*, *Ber.*, 1933, **66**, 943, 1080; 1936, **69**, 126.

Stigmastane (*Sitostane*)

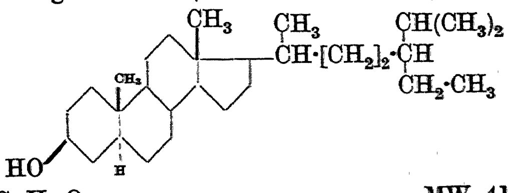


$C_{29}H_{52}$ MW, 400

Cryst. from EtOH. M.p. 85·4–85·7°. $[\alpha]_D^{20}$ + 25·4° in $CHCl_3$. Sol. Et_2O. Spar. sol. EtOH.

Windaus, Brunken, *Z. physiol. Chem.*, 1924, **140**, 47.
Bengtsson, *Z. physiol. Chem.*, 1935, **237**, 46.
Marker, Whittle, *J. Am. Chem. Soc.*, 1937, **59**, 2704.

Stigmastanol (*Fucostanol, sitostanol*)



$C_{29}H_{52}O$ MW, 416

Plates from EtOH. M.p. 136–7°. $[\alpha]_D^{20}$ + 24·8° in $CHCl_3$.

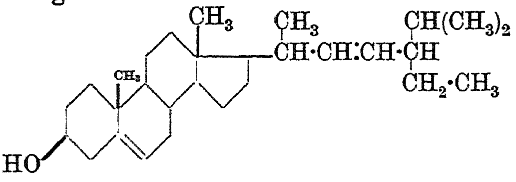
Acetyl: needles from EtOH. M.p. 130–1°. $[\alpha]_D^{20}$ + 15·3° in $CHCl_3$.

Benzoyl: plates from AcOEt. M.p. 135·5–137°.

3 : 5-*Dinitrobenzoyl*: plates from AcOEt. M.p. 214–15°. $[\alpha]_D^{20}$ + 13·1° in C_6H_6.

Coffey, Heilbron, Spring, *J. Chem. Soc.*, 1936, 738.
Windaus, Brunken, *Z. physiol. Chem.*, 1924, **140**, 47.
Bengtsson, *Z. physiol. Chem.*, 1935, **237**, 46.
Marker, Whittle, *J. Am. Chem. Soc.*, 1937, **59**, 2704.

Stigmasterol



$C_{29}H_{48}O$ MW, 412

Occurs in soya and calabar beans. Cryst. from EtOH. M.p. 170°. $[\alpha]_D^{22}$ − 51·0° in $CHCl_3$.

Acetyl: plates from EtOH. M.p. 144–144·6°. $[\alpha]_D^{20}$ − 55·5° in $CHCl_3$. *Tetrabromide*: plates from EtOH–$CHCl_3$. M.p. 211–12° (202–3°). $[\alpha]_D^{20}$ − 40°.

Propionyl: prisms from EtOH. M.p. 122°.

Palmityl: m.p. 99°.

Stearyl: m.p. 101°.

Benzoyl: prisms from $CHCl_3$–EtOH. M.p. 160°.

p-*Nitrobenzoyl*: needles from ligroin. M.p. 203°.

Anisoyl: m.p. 173·5–174°. $[\alpha]_D^{17}$ − 14·3°.

p-*Phenylazobenzoyl*: m.p. 191–2°.

p-*Toluenesulphonyl*: m.p. 148–50°. $[\alpha]_D$ − 47·1°.

Et ether: m.p. 98–100°. $[\alpha]_D^{28}$ − 38·5°.

n-*Butyl ether*: m.p. 109–10°. $[\alpha]_D^{26\cdot5}$ − 47·4°.

Benzyl ether: m.p. 118–19°. $[\alpha]_D^{26}$ − 39·5°.

Windaus, Hauth, *Ber.*, 1906, **39**, 4378.
Fernholz, *Ann.*, 1933, **508**, 215.
Guiteras, *Z. physiol. Chem.*, 1933, **214**, 89.
Fernholz, Chakravorty, *Ber.*, 1934, **67**, 2021.

α-Stilbazole (2-*Stilbazole*, 2-*styrylpyridine*, β-2-*pyridinostyrene*)

$C_{13}H_{11}N$ MW, 181

Cryst. from EtOH.Aq. M.p. 91°. B.p. 324–5°/750 mm., 194°/14 mm. Sol. Et_2O, CS_2. Mod. sol. EtOH, C_6H_6, ligroin. Insol. H_2O. Volatile in steam. $KMnO_4$ ⟶ benzoic + picolinic acids.

B,HCl,4H₂O: m.p. anhyd. 177°.

B,HAuCl₄: m.p. 185°.

B,HCl,HgCl₂: m.p. 181–3°.

B,H₂PtCl₆: m.p. 201°.

Picrate: m.p. 207°.

Methiodide: pale yellow cryst. from EtOH. M.p. 190–1°.

Wagstaff, *J. Chem. Soc.*, 1934, 277.
Harries, Lenart, *Ann.*, 1915, **410**, 95.
Chiang, Hartung, *J. Org. Chem.*, 1945, **10**, 21.

γ-Stilbazole (4-*Stilbazole*, 4-*styrylpyridine*, β-4-*pyridinostyrene*).

Prisms, m.p. 131°: leaflets from EtOH, m.p. 127° (130·5°). B.p. 208°/33 mm. Sol. EtOH, Et_2O, $CHCl_3$. Insol. H_2O.

Hydrochloride: m.p. 204°.

Hydrobromide: m.p. 221°.

Hydriodide: m.p. 174°.

B,HAuCl₄: m.p. 201°.

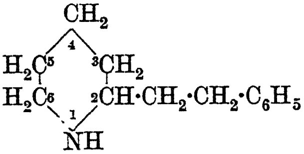
B₂,H₂PtCl₆: m.p. 310°.

Picrate: m.p. 113°.

Ethiodide: m.p. 179–80°.

Wagstaff, *J. Chem. Soc.*, 1934, 277.
Chiang, Hartung, *J. Org. Chem.*, 1945, **10**, 21.

α-Stilbazoline (2-*Phenylethylpiperidine*)



$C_{13}H_{19}N$ MW, 189

d-.

Liq. $[\alpha]_D^{18}$ + 12·16°.

Acid d-*tartrate*: rhombohedra. M.p. 50°.

l-.

Liq. $[\alpha]_D^{18}$ − 11·5°.

B,HCl: needles or prisms from Me_2CO. M.p. 116–17°.

Neutral l-*tartrate*: plates. M.p. 211–12°.

Acid d-*tartrate*: cryst. + H_2O. M.p. 78–80°.

dl-.

Liq. B.p. 288° (277–8°). D_4^0 0·9874. Misc. with EtOH, Et_2O, C_6H_6. Spar. sol. H_2O with strong alk. reaction.

B,HCl: needles from C_6H_6–pet. ether. M.p. 155°.

B,HBr: cryst. from C_6H_6. M.p. 173°.

B,HAuCl₄: yellow powder. M.p. 133–4°.

B₂,H₂PtCl₆: yellow powder. M.p. 187–9°.

Ladenburg, *Ber.*, 1904, **37**, 3688.
Baurath, *Ber.*, 1888, **21**, 822.
Bach, *Ber.*, 1901, **34**, 2233.

γ-Stilbazoline (*4-Phenylethylpiperidine*).
Oil. B.p. 200–10°/80 mm.
B,HAuCl₄ : red leaflets. M.p. 204° decomp.
B_2,H_2PtCl_6 : brown leaflets. M.p. about 210°.
Friedländer, *Ber.*, 1905, **38**, 2837.

Stilbene (sym.-*Diphenylethylene, toluylene, dibenzal, dibenzylidene*)

$C_6H_5{\cdot}CH$ ∥ $C_6H_5{\cdot}CH$ — *Cis* (isostilbene)

$C_6H_5{\cdot}CH$ ∥ $HC{\cdot}C_6H_5$ — *Trans* (stilbene)

$C_{14}H_{12}$ MW, 180

Cis :
Cryst. M.p. 5–6°. B.p. 148–9°/17 mm., 145°/13 mm., 136–7°/10 mm. n_D^{25} 1·6223.
Trans :
Cryst. from EtOH. M.p. 124°. B.p. 305°/720 mm., 166–7°/12 mm. Sol. Et_2O, C_6H_6. Shows blue fluor. Volatile in steam. Heat of comb. C_p 1765·7 Cal. n_D^{17} 1·6264.
Picrate : m.p. 94–5°.
$C_{14}H_{12},2SbCl_3$: m.p. 93°.
$C_{14}H_{12},C_6H_3(NO_2)_3$-1 : 3 : 5 : m.p. 115–20° (107–10°).

Böeseken, Elsen, *Rec. trav. chim.*, 1928, **47**, 694.
Schenck, Bergmann, *Ann.*, 1928, **463**, 112.
Späth, *Monatsh.*, 1914, **35**, 463.
Corson, B.P. 279,095, (*Chem. Abstracts*, 1928, **22**, 2755).
Ballard, Dehn, *J. Am. Chem. Soc.*, 1932, **54**, 3969.
Kharasch, Nudenberg, Fields, *J. Am. Chem. Soc.*, 1944, **66**, 1276.
Brackman, Plesch, *J. Chem. Soc.*, 1952, 2188.

Stilbene-α-carboxylic Acid.
See α-Phenylcinnamic Acid.

Stilbenediamine (α : β-*Diaminodibenzyl*, α : β-*diamino*-sym.-*diphenylethane, diphenylethylenediamine*)

$C_6H_5{\cdot}CH(NH_2){\cdot}CH(NH_2){\cdot}C_6H_5$

$C_{14}H_{16}N_2$ MW, 212

d-.
$[\alpha]_D + 134{\cdot}8°$.
Feist, Arnstein, *Ber.*, 1895, **28**, 3169.
l-.
$[\alpha]_D - 128°$.
dl-.
Cryst. from ligroin. M.p. 90–2°.
$B,HCl,2H_2O$: m.p. 251° decomp.
$B,H_2PtCl_6,2H_2O$: m.p. 222–5°.
Picrate : m.p. 220°.
N : N′-*Diacetyl* : m.p. above 360°.
N : N′-*Dibenzoyl* : m.p. 287°.
N : N′-*Dibenzylidene* : m.p. 152°.
Meso-.
Leaflets from Et_2O. M.p. 121°.
$B,2HCl,2H_2O$: m.p. 256° decomp.
B,2HBr : m.p. 276–8°.
B,H_2PtCl_6 : m.p. 265° decomp.
Picrate : m.p. 225°.
Dipicrate : m.p. 239°.
sym.-*Trinitrobenzene add. comp.* : m.p. 150°.
N : N′-*Diformyl* : m.p. 294°.
N-*Acetyl*-N′-*benzoyl* : m.p. 316°.
N : N′-*Dibenzoyl* : m.p. 350°.
N : N′-*Dibenzylidene* : m.p. 164°.

Biltz, Krebs, *Ann.*, 1912, **391**, 208.
Darapsky, Spannagel, *J. prakt. Chem.*, 1915, **92**, 289.

Stilbene dibromide (α : β-*Dibromo*-sym.-*diphenylethane*, α : β-*dibromodibenzyl*)

$C_6H_5{\cdot}CHBr{\cdot}CHBr{\cdot}C_6H_5$

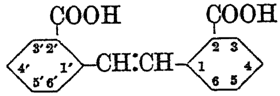
$C_{14}H_{12}Br_2$ MW, 340

(i) Needles. M.p. 237°. Sol. hot xylene. Mod. sol. Et_2O, CS_2. Spar. sol. EtOH.
(ii) Cryst. from hot EtOH. M.p. 111°. Sol. Et_2O. Spar. sol. EtOH.

Liebermann, *Ber.*, 1910, **43**, 1543.
Pfeiffer, *Ber.*, 1912, **45**, 1818.
Kato, *Chem. Abstracts*, 1932, **26**, 5260.
Ruggli, Lang, *Helv. Chim. Acta*, 1938, **31**, 47.
Reulos, *Compt. rend.*, 1943, **216**, 774.

Stilbene-α : β-dicarboxylic Acid.
See Diphenylfumaric Acid *and* Diphenylmaleic Acid.

Stilbene-2 : 2′-dicarboxylic Acid



$C_{16}H_{12}O_4$ MW, 268

Needles from dil. AcOH. M.p. 263–4° (rapid heat.), 250° (slow heat.).
Di-Et ester : $C_{20}H_{20}O_4$. MW, 324. Needles. M.p. 79–80°. Sol. Et_2O.

Hasselbach, *Ann.*, 1888, **243**, 258.

Stilbene-2 : 4′-dicarboxylic Acid.
Cryst. from AcOH. M.p. 295–6°.
Di-Et ester : needles from EtOH. M.p. 67–8°.

Hager, Arendonk, Shonle, *J. Am. Chem. Soc.*, 1944, **66**, 1982.

Stilbene-4 : 4′-dicarboxylic Acid.
Di-Me ester : $C_{18}H_{16}O_4$. MW, 296. Cryst. from EtOH. M.p. 226–7°. Insol. Et_2O. Sol. in EtOH has bluish-violet fluor.
Di-Et ester : needles. M.p. 130–1°.
Di-amidine HCl : cryst. + $2H_2O$. M.p. 355–60° decomp.

Meyer, Hofmann, *Monatsh.*, 1917, **38**, 358.
Hager, Arendonk, Shonle, *J. Am. Chem. Soc.*, 1944, **66**, 1982.
Oxley, Short, *J. Chem. Soc.*, 1948, 303.
Sah, *J. Am. Chem. Soc.*, 1942, **64**, 1482.

Stilbene dichloride (*α : β-Dichloro-*sym.*-diphenylethane, α : β-dichlorodibenzyl*)

$C_6H_5{\cdot}CHCl{\cdot}CHCl{\cdot}C_6H_5$

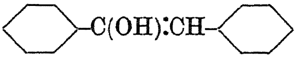
$C_{14}H_{12}Cl_2$ MW, 251

(i) Needles from EtOH. M.p. 191–3°. Sol. hot toluene. Spar. sol. EtOH.
(ii) Leaflets. M.p. 93–4°. Sol. ord. org. solvents. Sublimes.

Pfeiffer, Eistert, *J. prakt. Chem.*, 1930, **124**, 168.
Carré, Mauclère, *Compt. rend.*, 1931, **192**, 1567.
Pfeiffer, *Ber.*, 1912, **45**, 1816.
Reulos, *Compt. rend.*, 1943, **216**, 774.
Yura, Hashimoto, *J. Soc. Chem. Ind. Japan*, 1944, **47**, 814, (*Chem. Abstracts*, 1948, **42**, 6347).

Stilbene oxide.
See 1 : 2-Diphenylethylene oxide.

α-Stilbenol (*α-Hydroxystilbene, enol-deoxybenzoin*)



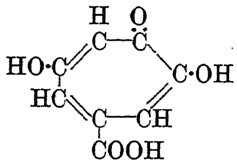
$C_{14}H_{12}O$ MW, 196

Free alcohol not stable.
Acetyl: pale yellow needles. M.p. 101°.

Barnes *et al.*, *J. org. Chem.*, 1943, **8**, 153.

Stilboestrol.
See Diethylstilboestrol.

Stipitatic Acid



$C_8H_6O_5$ MW, 182

Metabolic product of *Penicillium stipitatum*, Thom. M.p. 302–4° decomp.
Mono-Me deriv.: m.p. 273° decomp.
Di-Me deriv.: m.p. 163–5°.
Tri-Me derivs.: (i) m.p. 126–8°; (ii) m.p. 189–90°.
Diacetyl: (i) m.p. 172·5°; (ii) m.p. 176–8° decomp.

Birkinshaw, Chambers, Raistrick, *Biochem. J.*, 1942, **36**, 242.
Dewar, *Nature*, 1945, **155**, 50.
Corbett, Hassall, Johnson, Todd, *Chemistry and Industry*, 1949, 626.
Bartels-Keith, Johnson, Taylor, *J. Chem. Soc.*, 1951, 2352.

Stovaine (*1-Dimethylamino-2-methyl-2-butanol benzoate*)

$O{\cdot}CO{\cdot}C_6H_5$
$(CH_3)_2N{\cdot}CH_2{\cdot}C{\cdot}CH_2{\cdot}CH_3$
CH_3

$C_{14}H_{21}O_2N$ MW, 235

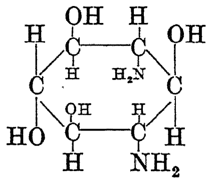
d-.
M.p. 186–7°. $[\alpha]_D^{20} + 8{\cdot}5°$.
l-.
M.p. 186–7°. $[\alpha]_D^{20} - 8{\cdot}5°$.
dl-.
M.p. 175°. Local anæsthetic.
B,HCl: cryst. from EtOH. M.p. 202°.
Picrate: cryst. from 50% EtOH.Aq. M.p. 115–16°.

Ribas, Rancaño, *Chem. Abstracts*, 1929, **23**, 2164.
Fourneau, Ribas, *Chem. Abstracts*, 1928, **22**, 761.
Fiore, *Chem. Zentr.*, 1916, I, 1076.
Riedel, D.R.P., 169,746, (*Chem. Zentr.*, 1906, I, 1584).

Note.—Both the free base and its hydrochloride are referred to as "Stovaine."

Stovarsol.
See under 2-Aminophenol-4-arsonic Acid.

Streptamine

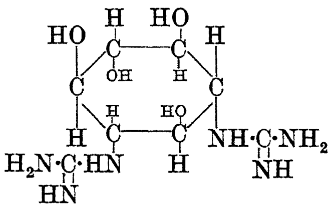


$C_6H_{14}O_4N_2$ MW, 178

Degradation product of streptomycin. M.p. above 290°. Optically inactive.
B,2HCl: m.p. above 280° decomp.
Dipicrate: m.p. above 300° decomp.
Hexa-acetyl: m.p. 245–8° decomp.
O-*Tetra-acetyl, HCl*: m.p. 260°.

Peck *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 776.
Wolfrom *et al.*, *J. Am. Chem. Soc.*, 1950, **72**, 1727.

Streptidine



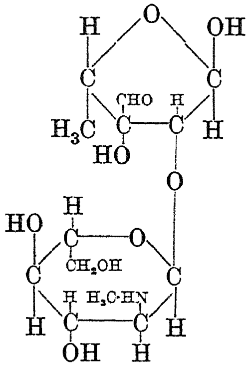
$C_8H_{18}O_4N_6$ MW, 262

Degradation product of streptomycin. It has no true m.p. Optically inactive.
B,2HCl: m.p. 170—210° decomp.
Dipicrate: m.p. 285°.
Dihelianthate: m.p. 240–50° decomp.

Octa-acetyl: m.p. 259–61°.

Peck *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 776.
Hooper *et al.*, *ibid.*, 2120.
Wolfrom *et al.*, *J. Am. Chem. Soc.*, 1950, **72**, 1727.

Streptobiosamine

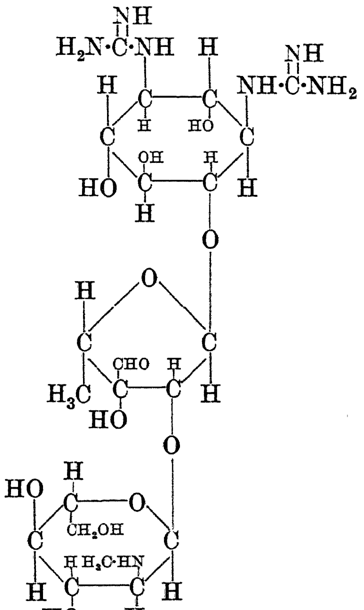


$C_{12}H_{21}O_8N$ MW, 307

Degradation product of streptomycin.
Tetra-acetyl: m.p. 188–9°.

Lemieux, Wolfrom, *Advances in Carbohydrate Chemistry*, 1948, Vol. 3, 337.
Kuehl *et al.*, *J. Am. Chem. Soc.*, 1949, **71**, 1445.

Streptomycin (N-*Methyl*-L-*glucosaminido-streptosido-streptidine*)

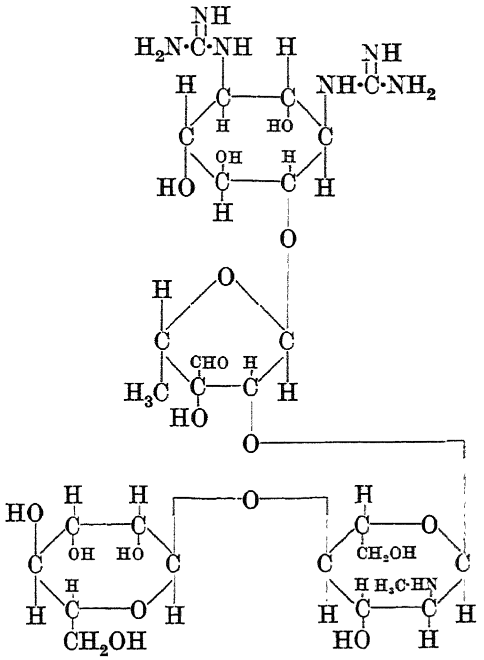


$C_{21}H_{39}O_{12}N_7$ MW, 581

Antibiotic obtained from *Streptomyces griseus*.
B,3HCl: $[\alpha]_D$ −86·7°. *Dihydro*: m.p. 190–5° decomp.
Helianthate: m.p. 220–6° decomp.
Reineckate: m.p. 162–4° decomp.

Schatz, Bugie, Waksman, *Proc. Soc. Exptl. Biol. Med.*, 1944, **55**, 66.
Lemieux, Wolfrom, *Advances in Carbohydrate Chemistry*, 1948, Vol. 3, p. 337.
Kuehl *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 1234.
Emery, *Chemistry and Industry*, 1952, 254 (*Review*).

Streptomycin B (*Mannosidostreptomycin*)



$C_{27}H_{49}O_{17}N_7$ MW, 743

Antibiotic occurring with streptomycin.
B,3HCl: amorphous powder. M.p. 190–200°. $[\alpha]_D^{25}$ − 47° in H_2O.
Reineckate: cryst. + $2H_2O$ from H_2O. M.p. 178–9° decomp.

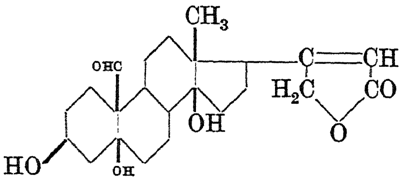
Fried, Staveley, *J. Am. Chem. Soc.*, 1949, **71**, 135; 1952, **74**, 5461.
Fried, Titus, *J. Am. Chem. Soc.*, 1948, **70**, 3615.

Streptothricin.

Antibiotic from *Streptomyces lavendulae*.
B,HCl: $[\alpha]_D^{25}$ − 51·3°.
Helianthate: m.p. 225–30°.
Reineckate: m.p. 192–4°.

Waksman, Woodruff, *Proc. Soc. Exptl. Biol. Med.*, 1942, **49**, 207.
Peck *et al.*, *J. Am. Chem. Soc.*, 1946, **68**, 772.

Strophanthidin (*Corchorin*)



$C_{23}H_{32}O_6$ MW, 404

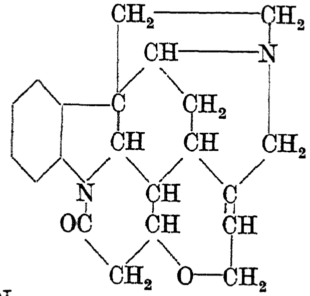
Occurs in *Strophanthus kombé*, Oliver. Leaflets + $2H_2O$ from H_2O. M.p. 169–70°, anhyd. 235°. Sol. Et_2O, $CHCl_3$, C_6H_6. Insol. pet. ether. $[\alpha]_D^{25}$ + 43·1° in MeOH.

Oxime : m.p. 270–5°.
Phenylhydrazone : m.p. 230–2°.
p-*Bromophenylhydrazone* : m.p. 200°.
Benzoyl : $[\alpha]_D^{26}$ + 47·8°.
p-*Bromobenzoyl* : m.p. 222–4°.

Lamb, Smith, *J. Chem. Soc.*, 1936, 444.
Jacobs, Elderfield, *J. Biol. Chem.*, 1935, **108**, 497.
Kon, *J. Soc. Chem. Ind.*, 1934, **53**, 956.
Gamble, Kon, *J. Chem. Soc.*, 1935, 443.
Elderfield, *J. Biol. Chem.*, 1936, **113**, 625.
Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., (Reinhold Publishing Corporation, New York), 516.

g-Strophanthin.
See Ouabain.

Strychnine



$C_{21}H_{22}O_2N_2$ MW, 334

Alkaloid occurring in seeds of *Strychnos nux vomica*, Linn., *S. ignatii*, Berg. Prisms from EtOH. M.p. 286–8° (270–1° slow heat.). B.p. 270°/5 mm. $[\alpha]_D^{18}$ − 139·3° in $CHCl_3$, − 104·5° in abs. EtOH. Sol. 6 parts $CHCl_3$, 66·6 parts Py, 165 parts C_6H_6, 6400 parts H_2O at 25°, and 12 parts boiling 90% EtOH. Prac. insol. abs. Et_2O. Cold sol. in H_2SO_4 + an oxidising agent, *e.g.* $K_2C_3O_7$, $KMnO_4$, PbO_2, MnO_2 ⟶ violet-blue col. ⟶ purple ⟶ red ⟶ yellow. Warm in dil. HNO_3 + $K_2Cr_2O_7$ ⟶ intense scarlet col. HNO_3 (D 1·4) ⟶ colourless sol. ⟶ yellow on heating. Bitter taste. Violent tetanic poison. Employed commercially in vermin killers.

B,HCl : needles + $1\frac{1}{2}H_2O$.
B,HBr : needles + H_2O. Spar. sol. H_2O.
B,HI : needles + H_2O. Spar. sol. H_2O.
B,HNO₃ : needles. Sol. 42 parts H_2O at 25°, 120 parts EtOH at 25°.
B₂,H₂SO₄ : needles + $5H_2O$. M.p. anhyd. 200°. Sol. 48 parts H_2O, 135 parts EtOH.
B₂,H₂Cr₂O₇ : orange-yellow needles. Sol. 1815 parts H_2O at 25°.
Monopicrate : decomp. at 270°.
Monopicrolonate : decomp. above 290°.
d-*Tartrate* : cryst. + $7H_2O$. M.p. 228°.
l-*Tartrate* : cryst. + $3\frac{1}{2}H_2O$. M.p. 242°.
Benzyl chloride : m.p. 303·5°.
Benzylidene deriv. : m.p. 235–7°.

Robinson, *Annual Report of Biochemistry*, 1935, **4**, 512 (*Bibl.*).
Leuchs, Beyer, *Ber.*, 1935, **68**, 290.
Clemo, *J. Chem. Soc.*, 1936, 1695.
Kotake, Mori, Mitsuwa, *Sci. Papers Inst. Phys. Chem. Research Tokyo*, 1937, **31**, 129.
Briggs, Openshaw, Robinson, *J. Chem. Soc.*, 1946, 903.
Chakravarti, Robinson, *Nature*, 1947, **160**, 18.
Woodward, *Nature*, 1948, **162**, 155.

ψ-Strychnine

$C_{21}H_{22}O_3N_2$ MW, 350

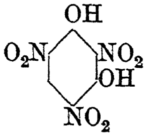
A strychnine alkaloid. Cryst. powder. M.p. 266–8° decomp. Mod. sol. $CHCl_3$. Spar. sol. Me_2CO, C_6H_6. Insol. Et_2O. $[\alpha]_D^{25}$ − 85·9° in $CHCl_3$, − 43·8° in abs. EtOH. H_2SO_4 + $K_2Cr_2O_7$ ⟶ violet col. Not bitter and is less toxic than strychnine. Zn + HCl ⟶ strychnine.

B,HCl : cryst. + $2H_2O$ from H_2O. $[\alpha]_D^{19}$ + 3·9° in H_2O, $[\alpha]_D^{24}$ + 8·3° in 80% EtOH.
B,HNO₃ : spar. sol. H_2O. $[\alpha]_D^{28}$ + 7·6° in 80% EtOH.
B,HClO₄ : needles from H_2O. Does not melt below 300°.
Ferrichloride : m.p. 234–5°.
Nitroso deriv. : cryst. from 80% EtOH. M.p. 292–4°. Sol. $CHCl_3$. Spar. sol. MeOH, EtOH, Et_2O, C_6H_6. $[\alpha]_D^{19}$ + 223·8° in $CHCl_3$.

Warnat, *Helv. Chim. Acta*, 1931, **14**, 1004.
Blount, Robinson, *J. Chem. Soc.*, 1932, 2305.

Stycerine.
See 1-Phenylglycerol.

Styphnic Acid (2 : 4 : 6-*Trinitroresorcinol*)



$C_6H_3O_8N_3$ MW, 245

Yellow cryst. from AcOEt. M.p. 179–80° (178°). Explodes on rapid heating. Sol. EtOH, Et_2O. Spar. sol. H_2O. Forms add. comps. with many hydrocarbons.

Di-Me ether: $C_8H_7O_8N_3$. MW, 273. Needles from EtOH. M.p. 124–5°.
Me-Et ether: $C_9H_9O_8N_3$. MW, 287. M.p. 92°.
Di-Et ether: $C_{10}H_{11}O_8N_3$. MW, 301. Leaflets from EtOH. M.p. 121°.

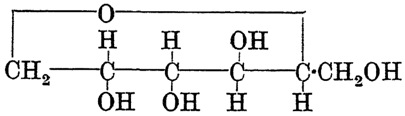
Brass, Fanta, *Ber.*, 1936, **69**, 6 (*Bibl.*).
Sah, *Chem. Abstracts*, 1932, **26**, 5927.
Vermeulen, *Rec. trav. chim.*, 1919, **38**, 107.
Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2043.

Stypticin.
See under Cotarnine.

Styptol.
See under Cotarnine.

Styracin.
See Cinnamyl cinnamate.

Styracitol (1 : 5-*Anhydro*-D-*mannitol*)

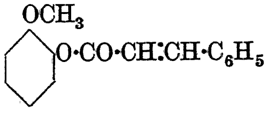


$C_6H_{12}O_5$ MW, 164

Occurs in fruit of *Styrax obassia*. Prisms from 90% EtOH. M.p. 157° (155°). Sol. H_2O. Spar. sol. cold EtOH. Prac. insol. Et_2O, Me_2CO, C_6H_6. Bitter-sweet taste. $[\alpha]_D^{20}$ — 71·72° in H_2O ($[\alpha]_D^{17}$ — 49·4° in H_2O). Does not reduce Fehling's.
Tetra-acetyl: prisms from H_2O, m.p. 66–7°: needles from EtOH, m.p. 58°. B.p. 200°/7 mm. $[\alpha]_D^{22}$ — 20·86° in EtOH.
Tetrabenzoyl: m.p. 142°.
Tetra-Me ether: b.p. 149–51°/24 mm., 143–4°/16 mm. D_4^{18} 1·1092. n_D^{14} 1·4516. $[\alpha]_D^{14}$ — 35·63°.
Diacetone deriv.: m.p. 96–7°. $[\alpha]_D^{17}$ — 115·24° in EtOH.
Dibenzylidene deriv.: (i) m.p. 163–5°. $[\alpha]_D^{25}$ — 148·73° in $CHCl_3$; (ii) m.p. 192–3°. $[\alpha]_D^{25}$ — 80·47° in $CHCl_3$.

Zervas, *Ber.*, 1930, **63**, 1689.
Freudenberg, Sheehan, *J. Am. Chem. Soc.*, 1940, **62**, 558.
Zervas, *Ber.*, 1940, **73**, 174.
Richtmeyer, Carr, Hudson, *J. Am. Chem. Soc.*, 1943, **65**, 1477.

Styracol (*Guaiacol cinnamate*)



$C_{16}H_{14}O_3$ MW, 254

Needles from EtOH. M.p. 130°.

Knoll, D.R.P., 62,176.

Styrene (*Styrol, styrolene, cinnamol, cinnamene, vinylbenzene, phenylethylene*)

$C_6H_5{\cdot}CH{:}CH_2$

C_8H_8 MW, 104

Occurs in storax. F.p. — 33°. M.p. — 33°. B.p. 145–145·8°, 52–3°/28 mm., 48°/20 mm., 40°/14 mm., 33°/10 mm. Sol. EtOH, Et_2O, MeOH, Me_2CO, CS_2. Spar. sol. H_2O. D_4^{20} 0·9090. n_D^{20} 1·5462.
Metastyrene: $(C_8H_8)_x$. MW, $(104)_x$. Amorphous mass. Spar. sol. hot Et_2O. Insol. H_2O, EtOH. Irradiation in C_6H_6 or dist. ⟶ styrene.
Polystyrene: a thermoplastic rigid polymer.
Tetrastyrene: $C_{32}H_{32}$. MW, 416. B.p. 210–17°/0·05 mm.

Naugatuck, D.R.P., 600,268, (*Chem. Abstracts*, 1934, **28**, 6448).
Sontag, *Ann. chim.*, 1934, I, 359.
Zal'kind, Bulavskii, *Chem. Abstracts*, 1936, **30**, 1368.
Gibbons, Smith, U.S.P., 1,938,827, (*Chem. Abstracts*, 1934, **28**, 1367).
Houtz, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 1609.
Gautier, Gautier, *Bull. soc. chim.*, 1933, **53**, 323.
Midgley, Henne, Leicester, *J. Am. Chem. Soc.*, 1936, **58**, 1961.
Graves, U.S.P., 2,036,410, (*Chem. Abstracts*, 1936, **30**, 3446).
Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 1133 (*Bibl.*).
I.G., U.S.P., 2,005,295, (*Chem. Abstracts*, 1935, **29**, 5203).
Staudinger, D.R.P., 610,478, (*Chem. Abstracts*, 1935, **29**, 3752).
Hibbert, Burt, *J. Am. Chem. Soc.*, 1925, **47**, 2240.

Styrene bromohydrin (*Phenylethylene bromohydrin, β-bromo-α-hydroxyethylbenzene, bromomethylphenylcarbinol*)

$C_6H_5{\cdot}CH(OH){\cdot}CH_2Br$

C_8H_9OBr MW, 201

B.p. 109–10°/2 mm. n_D^{17} 1·5800. D_4^{20} 1·4994.
Acetyl: b.p. 105–7°/3 mm. D_{20}^{20} 1·386. n_D^{20} 1·5380.

Read, Reid, *J. Chem. Soc.*, 1928, 1488.
Marvel, Moon, *J. Am. Chem. Soc.*, 1940, **62**, 45.

Styrene chlorohydrin (*Phenylethylene chlorohydrin, β-chloro-α-hydroxyethylbenzene, chloromethylphenylcarbinol*)

$C_6H_5{\cdot}CH(OH){\cdot}CH_2Cl$

C_8H_9OCl MW, 156·5

B.p. 110–11°/6 mm. $D_4^{20.5}$ 1·1646. n_D^{20} 1·5400.
Acetyl: oil. B.p. 101–4°/3 mm. n_D^{12} 1·5182.
p-*Nitrobenzoyl*: cryst. from EtOH. M.p. 81°.

Detoeuf, *Bull. soc. chim.*, 1922, **31**, 176.
Emerson, *J. Am. Chem. Soc.*, 1945, **67**, 516.
Hanby, Rydon, *J. Chem. Soc.*, 1946, 114.

Styrene dibromide (α : β-*Dibromoethylbenzene, phenylethylene dibromide*)

$C_6H_5·CHBr·CH_2Br$

$C_8H_8Br_2$ MW, 264

Leaflets or needles from EtOH.Aq. M.p. 74–74·5° (72–3°). B.p. 139–41°/15 mm. Sol. EtOH, Et_2O, C_6H_6, AcOH, ligroin.

Evans, Morgan, *J. Am. Chem. Soc.*, 1913, **35**, 56.
Glaser, *Ann.*, 1870, **154**, 154.
Zincke, *Ann.*, 1883, **216**, 288.

Styrene dichloride (α : β-*Dichloroethylbenzene, phenylethylene dichloride*)

$C_6H_5·CHCl·CH_2Cl$

$C_8H_8Cl_2$ MW, 175

B.p. 233–4°/759 mm., 114·5–115·5°/15 mm. D_4^{15} 1·240. n_D^{15} 1·5544.

Biltz, *Ann.*, 1897, **296**, 275.

Styrene Glycol (*Phenylethylene glycol*, α : β-*dihydroxyethylbenzene*)

$C_6H_5·CH(OH)·CH_2OH$

$C_8H_{10}O_2$ MW, 138

Needles from ligroin. M.p. 67–8°. B.p. 272–4°/755 mm. Very sol. H_2O, EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Spar. sol. ligroin.

Me ether: $C_9H_{12}O_2$. MW, 152. B.p. 237–8°, 132°/18 mm. D^0 1·080.

Et ether: $C_{10}H_{14}O_2$. MW, 166. B.p. 242–3°. D^0 1·054.

Di-Et ether: $C_{12}H_{18}O_2$. MW, 194. B.p. 105–6°/10 mm. Dil. $H_2SO_4 \longrightarrow$ phenylacetaldehyde.

Diformyl: b.p. 164–5°/25 mm. D^0 1·2091.

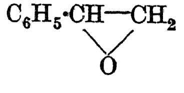
Diacetyl: b.p. 274°/755 mm., 183–5°/25 mm.

Dibenzoyl: needles from EtOH or toluene. M.p. 96–7°. Sublimes.

β-*Acetyl*: b.p. 136–7°/6 mm. D_{20}^{20} 1·1438. n_D^{20} 1·5260.

Zincke, *Ann.*, 1883, **216**, 293.
Tiffeneau, *Compt. rend.*, 1907, **145**, 812.
Späth, *Monatsh.*, 1914, **35**, 332.
Balla, *Compt. rend.*, 1934, **198**, 948.
Milas, Sussman, *J. Am. Chem. Soc.*, 1937, **59**, 2345.
Kindler, Blaas, *Ber.*, 1944, **77**, 585.

Styrene oxide (*Phenylethylene oxide*)



C_8H_8O MW, 120

Liq. with aromatic odour. B.p. 191–2°, 84–5°/15 mm., 77·5–78·5°/12 mm. D_4^{16} 1·0523.

Fourneau, Tiffeneau, *Compt. rend.*, 1905, **140**, 1595; 1908, **146**, 697.
Hibbert, Burt, *Organic Syntheses*, Collective Vol. I, 481.
Read, Reid, *J. Chem. Soc.*, 1928, 1487.
Emerson, *J. Am. Chem. Soc.*, 1945, **67**, 516.

Styrol.
See Styrene.

Styrolene.
See Styrene.

Styrone.
See Cinnamaldehyde.

Styrylacetic Acid (3-*Phenylvinylacetic acid*, 2-*benzylidenepropionic acid*)

$C_6H_5·CH{:}CH·CH_2·COOH$

$C_{10}H_{10}O_2$ MW, 162

I.

Needles from H_2O. M.p. 87°. B.p. 302°. Sol. EtOH, Et_2O, hot CS_2.

K salt: m.p. 245° decomp.

Me ester: $C_{11}H_{12}O_2$. MW, 176. B.p. 185°/about 20 mm.

Et ester: $C_{12}H_{14}O_2$. MW, 190. B.p. 281–2°/763 mm., 184°/44 mm., 164–5°/25 mm. $D_4^{20·5}$ 1·0340. $n_D^{20·5}$ 1·53271.

Chloride: $C_{10}H_9OCl$. MW, 180·5. M.p. 41–2°. B.p. 106–8°/0·4 mm.

Amide: $C_{10}H_{11}ON$. MW, 161. Needles from C_6H_6. M.p. 130°. Sol. EtOH, $CHCl_3$, hot H_2O. Spar. sol. Et_2O.

Anhydride: $C_{20}H_{18}O_3$. MW, 306. M.p. 120–1°.

Nitrile: cinnamyl cyanide. $C_{10}H_9N$. MW, 143. Leaflets from pet. ether. M.p. 61–2°.

II. *Allo-*.

Amide: needles from C_6H_6–pet. ether. M.p. 85–6°.

Stoermer, Stockmann, *Ber.*, 1914, **47**, 1794.
Borsche, Niemann, Hartman, *Ber.*, 1936, **69**, 1996.
Linstead, Williams, *J. Chem. Soc.*, 1926, 2741.
Fichter, Pfister, *Ber.*, 1904, **37**, 2001.

2-Styrylacrylic Acid (*Cinnamylideneacetic acid*, 4-*phenyl*-1 : 3-*butadiene*-1-*carboxylic acid*, 4-*phenylvinylacrylic acid*)

$C_6H_5·CH{:}CH·CH{:}CH·COOH$

$C_{11}H_{10}O_2$ MW, 174

α-.

Plates from EtOH, prisms from C_6H_6. M.p. 165–6°. Sol. EtOH. Spar. sol. pet. ether. Heat of comb. C_p 1312·7 Cal, C_v 1311·8 Cal.

A,$CH_3·NH_2$: m.p. 142–3°.

A,$C_2H_5·NH_2$: m.p. 117–18°.

A,$(C_2H_5)_2NH$: m.p. about 80°.

Me ester: $C_{12}H_{12}O_2$. MW, 188. M.p. 71°. B.p. 185°/20 mm.

Et ester: $C_{13}H_{14}O_2$. MW, 202. Oil. $D^{79·8}$ 0·9985.

Isopropyl ester: $C_{14}H_{16}O_2$. MW, 216. Yellow oil. B.p. 169°/9 mm. D_4^{18} 1·0256. $n_D^{13·7}$ 1·6066.

Amide: $C_{11}H_{11}ON$. MW, 173. Leaflets from C_6H_6. M.p. 185°. *Acetyl*: m.p. 177–8°.

Methylamide: $C_{12}H_{13}ON$. MW, 187. Needles from C_6H_6–EtOH. M.p. 157°.

Ethylamide: $C_{13}H_{15}ON$. MW, 201. Needles from C_6H_6. M.p. 143–4°.

Diethylamide: $C_{15}H_{19}ON$. MW, 229. M.p. 106°.

Anhydride: $C_{22}H_{18}O_3$. MW, 330. Needles from AcOH. M.p. 152°.

Nitrile: $C_{11}H_9N$. MW, 155. Cryst. from ligroin. M.p. 42°. B.p. 285°, 159°/285 mm.

β-. *Allo-*.

Needles from C_6H_6. M.p. 138°. Sol. Et_2O, CS_2, $CHCl_3$, hot C_6H_6. Heat of comb. C_p 1321·6 Cal, C_v 1320·7 Cal.

Me ester: f.p. — 15°. Decomp. on dist.

Riiber, *Ber.*, 1911, **44**, 2390; *Ber.*, 1904, **37**, 2274.
Reynolds, *Am. Chem. J.*, 1911, **46**, 200.
Stobbe, *Ber.*, 1912, **45**, 3408.
English, Gregory, *J. Am. Chem. Soc.*, 1947, **69**, 2123.
Pandya *et al.*, *J. Indian. Chem. Soc.*, 1947, **24**, 437, 443.

Styryl Alcohol.
See Cinnamyl Alcohol.

Styrylamine.
See Cinnamylamine.

Styryl bromide.
See β-Bromostyrene.

1-Styryl-1-butylene.
See 1-Phenyl-1 : 3-hexadiene.

4-Styryl-1-butylene.
See 1-Phenyl-1 : 5-hexadiene.

Styryl chloride.
See β-Chlorostyrene.

Styryl cyanide.
See under Cinnamic Acid.

2-Styryl-1-cyanoacrylic Acid.
See Cinnamylidenecyanoacetic Acid.

Styrylglycollic Acid (*1-Hydroxy-3-phenylvinylacetic acid, 1-hydroxy-2-benzylidenepropionic acid, benzylidene-lactic acid*)

C_6H_5·CH:CH·CH(OH)·COOH

$C_{10}H_{10}O_3$ MW, 178

Needles from H_2O. M.p. 137°. Very sol. boiling H_2O. Spar. insol. H_2O, Et_2O. Insol. CS_2, C_6H_6, ligroin.

Amide: $C_{10}H_{11}O_2N$. MW, 177. Plates from H_2O. M.p. 141·5°. Very sol. EtOH. Sol. hot $CHCl_3$, C_6H_6. Spar. sol. Et_2O, CS_2.

Nitrile: cinnamaldehyde cyanhydrin. $C_{10}H_9ON$. MW, 159. Plates from CS_2. M.p. 74°. *Benzoyl*: cryst. from H_2O. M.p. 72–3°.

Acetyl: needles from H_2O. M.p. 79° (+ $1H_2O$), 90–1° (anhyd.). Very sol. most org. solvents.

Fittig, Ginsberg, Petkow, *Ann.*, 1898, **299**, 20.
Thiele, Sulzberger, *Ann.*, 1901, **319**, 208.
Bougault, *J. pharm. chim.*, 1913, **8**, 404.

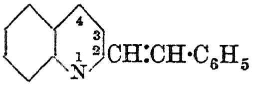
6-Styryl-α : γ-lutidine.
See 4 : 6-Dimethyl-α-stilbazole.

Styrylphenol.
See Hydroxystilbene.

2-Styrylpropionic Acid.
See 3-Benzylidenebutyric Acid.

1-Styrylpyridine.
See Stilbazole.

2-Styrylquinoline (*2-Benzylidenequinaldine, sym.-phenyl-2-quinolylethylene, α-irazole*)



$C_{17}H_{13}N$ MW, 231

Needles from Et_2O–EtOH. M.p. 99–100°. Sol. $CHCl_3$, CS_2, hot EtOH. Insol. H_2O. $CrO_3 \longrightarrow$ benzoic + quinaldinic acids.

B, Maleic acid: $C_{21}H_{17}O_4N$. MW, 347. Silky needles from butyl acetate. M.p. 165–7°.

Ismailsky, *J. prakt. Chem.*, 1912, **85**, 91.
Noelting, Witte, *Ber.*, 1906, **39**, 2750.
Bergmann, *J. Am. Chem. Soc.*, 1938, **60**, 2811.

4-Styrylquinoline (*ω-Benzylidenelepidine, sym.-phenyl-4-quinolylethylene*).

Cryst. from EtOH. M.p. 92°. Sol. Et_2O. Spar. sol. H_2O. $CrO_3 \longrightarrow$ cinchoninic acid.

Doebner, Miller, *Ber.*, 1885, **18**, 1646.

Suberamic Acid.
See under Suberic Acid.

Suberane.
See Cycloheptane.

Suberanilic Acid.
See under Suberic Acid.

Suberene.
See Cycloheptene.

Suberic Acid (*Hexane-1 : 6-dicarboxylic acid*)

HOOC·CH_2·CH_2·CH_2·CH_2·CH_2·CH_2·COOH

$C_8H_{14}O_4$ MW, 174

Needles from H_2O. M.p. 144° (139–40°). B.p. 279°/100 mm., 258·5°/50 mm., 230°/15 mm., 219·5°/10 mm. Spar. sol. H_2O, Et_2O. Insol. $CHCl_3$. Heat of comb. C_p 982·8 Cal., C_v 988·6 Cal. k (first) = $2·58 \times 10^{-5}$ at 25°; (second) = $2·5 \times 10^{-6}$ at 100°.

Di-brucine salt: m.p. 102°.

Me ester: $C_9H_{16}O_4$. MW, 188. M.p. 14–15°. B.p. 185–6°/18 mm. D^{20} 1·047.

Di-Me ester: $C_{10}H_{18}O_4$. MW, 202. F.p. — 3·1°. B.p. 268°, 174–5°/13–20 mm., 130–1°/9 mm. D_4^{20} 1·0217. n_D^{20} 1·43408.

Et ester: $C_{10}H_{18}O_4$. MW, 202. M.p. 21–2° (25°). B.p. 186–188·5°/16 mm. D^{23} 1·037. n_D^{23} 1·4412. *Chloride*: $C_{10}H_{17}O_3Cl$. MW, 220·5. B.p. 132°/15 mm. p-*Toluidide*: m.p. 74°.

Di-Et ester: $C_{12}H_{22}O_4$. MW, 230. F.p. 5·9°. B.p. 282°/763 mm., 251–3°/320 mm. D_4^{20} 0·9822. n_D^{20} 1·43278.

Phenacyl ester: $C_{16}H_{20}O_5$. MW, 292. M.p. 102·4°.
p-*Bromophenacyl ester*: $C_{16}H_{19}O_5Br$. MW, 371. M.p. 144·2°.
Dichloride: $C_8H_{12}O_2Cl_2$. MW, 211. B.p. 162–3°/15 mm., 149–50°/12 mm. $D_4^{20·8}$ 1·1718. $n_D^{20·6}$ 1·46847.
Amide: suberamic acid. $C_8H_{15}O_3N$. MW, 173. Cryst. from H_2O. M.p. 125–7°.
Diamide: $C_8H_{16}O_2N_2$. MW, 172. Cryst. from H_2O. M.p. 216–17°.
Anhydride: suberic anhydride. $C_8H_{12}O_3$. MW, 156. Needles from C_6H_6. M.p. 65–6° (63°).
Dinitrile: suberonitrile. $C_8H_{12}N_2$. MW, 136. M.p. — 3·5°. B.p. 185°/15 mm., 176–8°/11 mm.
Anilide: suberanilic acid. M.p. 128°.
Dianilide: m.p. 187° (183°).
Di-p-*toluidide*: m.p. 218°.
Dihydrazide: m.p. 185–6°.

Green, Hilditch, *J. Chem. Soc.*, 1937, 766.
Rozanov, Belikov, *Chem. Abstracts*, 1930, **24**, 3765.
Verkade, *Rec. trav. chim.*, 1927, **46**, 137.
Arppe, *Ann.*, 1862, **124**, 89.
Dale, Schorlemmer, *Ann.*, 1879, **199**, 145.
Blaise, Koehler, *Bull. soc. chim.*, 1909, **5**, 690.
Kenner, Morton, *Ber.*, 1939, **72**, 456.

Suberol.
See Cycloheptanol.
Suberone.
See Cycloheptanone.
Suberoxime.
See under Cycloheptanone.
Suberyl Alcohol.
See Cycloheptanol.
Suberylamine.
See Cycloheptylamine.
Suberylene.
See Cycloheptene.
Succinaldehydic Acid.
See 2-Aldehydopropionic Acid.

Succinamic Acid (*Succinic acid monoamide*)

$CH_2·CONH_2$
$CH_2·COOH$

$C_4H_7O_3N$ MW, 117

Needles from Me_2CO. M.p. 157° (154°). Mod. sol. H_2O, hot Me_2CO. Spar. sol. EtOH, C_6H_6, ligroin. Heat at 200° ⟶ succinimide.
A,NH₂·NH₂: m.p. 113°.
Me ester: $C_5H_9O_3N$. MW, 131. Plates. M.p. 89–91°. Sol. H_2O, EtOH, Me_2CO. Spar. sol. Et_2O, pet. ether.
Et ester: $C_6H_{11}O_3N$. MW, 145. M.p. 74–5°. B.p. 230–40°.
Amide: *see* Succinamide.

Rubtzov, *Chem. Abstracts*, 1924, **18**, 1472.
Jeffery, Vogel, *J. Chem. Soc.*, 1934, 1103.

Succinamide (*Succinic acid diamide*)

$CH_2·CO·NH_2$
$CH_2·CO·NH_2$

$C_4H_8O_2N_2$ MW, 116

Needles from H_2O. M.p. 260° decomp. (242·3°). Sol. hot H_2O. Insol. EtOH, Et_2O. Heat of comb. C_p 509·7 Cal. Heat ⟶ succinimide.
N-d-*Glucoside*: $C_{10}H_{18}O_7N_2$. MW, 278. Prisms + $2H_2O$ from EtOH.Aq. M.p. 88–90° (192° decomp., anhyd.). $[\alpha]_D^{19}$ − 17·40° in H_2O.

Morrell, *J. Chem. Soc.*, 1914, **105**, 2705.

Succinanil (N-*Phenylsuccinimide*)

$CH_2·CO$
$\quad\quad\quad >N·C_6H_5$
$CH_2·CO$

$C_{10}H_9O_2N$. MW, 175

Needles from H_2O. M.p. 156°. Distils at about 400° without decomp. Sol. Et_2O, hot EtOH. Insol. H_2O.

Warren, Briggs, *Ber.*, 1931, **64**, 29.
Ruggli, *Ann.*, 1916, **412**, 4.

Succinanilic Acid (*Succinic acid monoanilide*)

$CH_2·CO·NH·C_6H_5$
$CH_2·COOH$

$C_{10}H_{11}O_3N$ MW, 193

Needles from hot H_2O. M.p. 144·5–145·5° (148·5°). Sol. EtOH, Et_2O, hot H_2O. Heat of comb. C_p 1166·5 Cal. $k = 2·03 \times 10^{-5}$ at 25°. Heat ⟶ succinanil.
A,CH₃·NH₂: m.p. 115–20°.
Me ester: $C_{11}H_{13}O_3N$. MW, 207. Needles from EtOH. M.p. 97–8°.
Et ester: $C_{12}H_{15}O_3N$. MW, 221. Cryst. from Et_2O–pet. ether. M.p. 56·5–57·5°.
Amide: $C_{10}H_{12}O_2N_2$. MW, 192. Needles from H_2O. M.p. 181°. Heat of comb. C_p 1244·6 Cal.
Anilide: *see* Succinanilide.

Menschutkin, *Ann.*, 1872, **162**, 176.
See also Warren, Briggs, *Ber.*, 1931, **64**, 29.

Succinanilide (*Succinic acid dianilide*, N:N′-*diphenylsuccinamide*)

$CH_2·CO·NH·C_6H_5$
$CH_2·CO·NH·C_6H_5$

$C_{16}H_{16}O_2N_2$ MW, 268

Needles from EtOH. M.p. 230°. Sol. Et_2O, hot EtOH. Insol. H_2O. Heat. of comb C_p 1971·3 Cal. Dist. ⟶ succinanil + aniline.

Morrell, *J. Chem. Soc.*, 1914, **105**, 1736, 2702.
Mistry, Guha, *J. Indian Chem. Soc.*, 1930, I, 797.

Succindialdehyde (*Succinic dialdehyde, succinic aldehyde, dialdehydoethane*)

$CH_2{\cdot}CHO$
$CH_2{\cdot}CHO$

$C_4H_6O_2$ MW, 86

Only polymers produced in preparation. Heated in vacuo ⟶ monomer. B.p. 169–70° decomp., 56·5°/9 mm. D_4^{18} 1·069. n_D^{18} 1·42617.

Monoxime: *Me ether*, b.p. 67°/10 mm.

Dioxime: m.p. 173°.

Di-[di-Me]-acetal: b.p. 201–2°/772 mm., 99°/22 mm., 89°/13 mm. n_D^{20} 1·41555.

Di-[di-Et]-acetal: b.p. 210–15° decomp., 137°/35 mm., 116°/20 mm.

Polymer: m.p. 65°. B.p. 169°.

Harries, *Ann.*, 1913, **395**, 260.
Harries, Hohenemser, *Ber.*, 1908, **41**, 255 (*Footnote* 4).
Schöpf, Lehmann, *Ann.*, 1935, **518**, 1.

Succinic Acid (*Ethane*-1:2-*dicarboxylic acid*)

$CH_2{\cdot}COOH$
$CH_2{\cdot}COOH$

$C_4H_6O_4$ MW, 118

Occurs in fossils, algæ, lichens, fungi, etc. Prisms from H_2O. M.p. 185° (184·5–185°). B.p. 235° ⟶ anhydride. Sol. EtOH, MeOH, Me_2CO, hot H_2O, hot H·COOH. Spar. sol. Et_2O. k (first) = 6·37 × 10^{-3} (6·6 × 10^{-5}) at 25°; (second) = 2·54 × 10^{-6} (2·7 × 10^{-6}) at 25°.

A,2NH₂OH: m.p. 121° decomp.

Brucine salt: m.p. 216–18°. $[\alpha]_D^{20}$ − 25·4° in H_2O.

Quinine salt: m.p. 198–200°. $[\alpha]_D^{20}$ − 165·5° in H_2O.

Strychnine salt: m.p. 210°. $[\alpha]_D^{20}$ − 25·6° in H_2O.

Me ester: $C_5H_8O_4$. MW, 132. M.p. 58°. B.p. 151°/20 mm., 121–3°/4 mm.

Di-Me ester: *see* Dimethyl succinate.

Et ester: $C_6H_{10}O_4$. MW, 146. M.p. 8°. B.p. 172°/42 mm., 146–9°/17 mm., 119°/3 mm. D^{20} 1·1466. n_D^{20} 1·4327. *Chloride*: $C_6H_9O_3Cl$. MW, 164·5. B.p. 144°/90 mm., 92°/20 mm.

Me-Et ester: $C_7H_{12}O_4$. MW, 160. F.p. below − 20°. B.p. 208·2°.

Di-Et ester: *see* Diethyl succinate.

Di-2-hydroxyethyl ester: $C_8H_{14}O_6$. MW, 206. B.p. 176–80°/0·01 mm. p-*Nitrobenzoyl deriv.*: m.p. 90–1°. *Diphenylurethane*: m.p. 113°.

Di-2-chloroethyl ester: $C_8H_{12}O_4Cl_2$. MW, 243. B.p. 204–5°/30 mm.

Propyl ester: $C_7H_{12}O_4$. MW, 160. M.p. 15°. B.p. 126°/3 mm. D^{20} 1·1071. n_D^{20} 1·4343.

Dipropyl ester: $C_{10}H_{18}O_4$. MW, 202. M.p. − 10·4°. B.p. 250·8° (246–7°/762·2 mm.). D^{20} 1·0011. n_D^{20} 1·4252.

Di-isopropyl ester: b.p. 228°/761 mm.

Butyl ester: $C_8H_{14}O_4$. MW, 174. M.p. 8·6°. B.p. 136·5°/3 mm. D^{20} 1·0732. n_D^{20} 1·4360.

Dibutyl ester: $C_{12}H_{22}O_4$. MW, 230. M.p. − 29·25° (− 37·5°). B.p. 274·5°, 145°/4 mm. D^{20} 0·9652. n_D^{20} 1·4369.

Di-sec. n-*butyl ester*: b.p. 255·5–256·5°/750 mm. D_4^{20} 0·9735.

Di-isobutyl ester: b.p. 264·8–265·8°.

n-*Amyl ester*: $C_9H_{16}O_4$. MW, 188. M.p. 17·2°. B.p. 147°/3 mm. D^{20} 1·0460. n_D^{20} 1·4378.

Di-n-amyl. ester: $C_{14}H_{26}O_4$. MW, 258. M.p. − 9°. B.p. 171·5°/16 mm.

Di-active-*amyl ester*: b.p. 175°/20 mm.

Di-isoamyl ester: b.p. 298–9°/765·4 mm., 289·9°/728 mm.

Di-n-heptyl ester: $C_{18}H_{34}O_4$. MW, 314. B.p. 350–1°.

Dihexadecyl ester: $C_{36}H_{70}O_4$. MW, 566. M.p. 58°.

Diallyl ester: $C_{10}H_{14}O_4$. MW, 198. B.p. 249–50°/757·3 mm.

Cyclohexyl ester: $C_{10}H_{16}O_4$. MW, 200. M.p. 44°.

d-*Menthyl ester*: $C_{14}H_{24}O_4$. MW, 256. $[\alpha]_D$ + 55·68° in C_6H_6. *Cinchonidine salt*: m.p. 141–3°. $[\alpha]_D$ − 46·72° in EtOH.

l-*Menthyl ester*: m.p. 64°. $[\alpha]_D$ − 64·00° in $CHCl_3$.

Di-l-*menthyl ester*: $C_{24}H_{42}O_4$. MW, 394. M.p. 63°. $[\alpha]_D^{20}$ − 82·4° in $CHCl_3$.

l-*Bornyl ester*: $C_{14}H_{22}O_4$. MW, 254. M.p. 58°. $[\alpha]_D$ − 35·94° in EtOH.

Di-l-*bornyl ester*: $C_{24}H_{38}O_4$. MW, 390. M.p. 83·7°. $[\alpha]_D$ − 42·39° in EtOH.

Phenyl ester: $C_{10}H_{10}O_4$. MW, 194. M.p 98°.

Diphenyl ester: $C_{16}H_{14}O_4$. MW, 270. M.p. 121° (118°). B.p. 222·5°/15 mm.

Di-o-nitrophenyl ester: $C_{16}H_{12}O_8N_2$. MW, 360. M.p. 162°.

Di-m-nitrophenyl ester: m.p. 153°.

Di-p-nitrophenyl ester: m.p. 178°.

Di-o-tolyl ester: $C_{18}H_{18}O_4$. MW, 298. B.p. 238–40°/5 mm.

Di-m-tolyl ester: m.p. 60°.

Di-p-tolyl ester: m.p. 121°.

Di-1-naphthyl ester: $C_{24}H_{18}O_4$. MW, 370. M.p. 155°.

Di-2-naphthyl ester: m.p. 163°.

Benzyl ester: $C_{11}H_{12}O_4$. MW, 208. M.p. 46·5–47° (59°).

Dibenzyl ester: $C_{18}H_{18}O_4$. MW, 298. M.p. 45° (49–50°). B.p. 238°/14 mm.

Amide: *see* Succinamic Acid.

Diamide: *see* Succinamide.

Imide: *see* Succinimide.

Chloride: *see* Succinyl chloride.

Bromide: *see* Succinyl bromide.

Anhydride: *see* Succinic Anhydride.

Nitrile: *see* 2-Cyanopropionic Acid.

Dinitrile: *see* Succinonitrile.

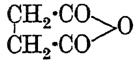
Et ester-nitrile: *see under* 2-Cyanopropionic Acid.

Amide-nitrile: *see under* 2-Cyanopropionic Acid.
Anilide: *see* Succinanilic Acid.
Dianilide: *see* Succinanilide.
Di-2 : 4-*dichloroanilide*: m.p. 248°.
Mono-o-*nitroanilide*: m.p. 132–132·5°.
Mono-m-*nitroanilide*: m.p. 181–2°.
Mono-p-*nitroanilide*: m.p. 196–7°.
Di-p-*nitroanilide*: m.p. 260°.
Me-anilide: m.p. 91–92·5°.
Di-Me-anilide: m.p. 156·5°.
Et-anilide: m.p. 92–3°.
Di-Et-anilide: m.p. 106°.
o-*Toluidide*: m.p. 97°.
Di-o-*toluidide*: m.p. 100°.
p-*Toluidide*: m.p. 179–80°.
Di-p-*toluidide*: m.p. 256°.
1-*Naphthalide*: m.p. 171°.
Di-1-*naphthalide*: m.p. 285° decomp.
2-*Naphthalide*: m.p. 190–2°. *Et ester*: m.p. 99–100°.
Di-2-naphthalide: m.p. 266°.
Peroxide: *see* Succinperoxide.

Leffler, Adams, *J. Am. Chem. Soc.*, 1936, **58**, 1553.
I.G., D.R.P., 485,313, (*Chem. Abstracts*, 1930, **24**, 862).
Fourneau, Sabetay, *Bull. soc. chim.*, 1929, **45**, 834.
Zaidan, Kenkyuja, D.R.P., 469,234, (*Chem. Abstracts*, 1929, **23**, 2110).
Conzen-Crowet, *Bull. soc. chim. Belg.*, 1926, **35**, 165.

Succinic Aldehyde.
See Succindialdehyde.

Succinic Anhydride (2 : 5-*Diketotetrahydrofuran*)



$C_4H_4O_3$ MW, 100

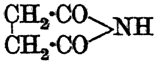
Cryst. from $CHCl_3$. M.p. 119·3–119·6°. B.p. 261°, 189°/100 mm., 169°/50 mm., 139°/15 mm., 131°/10 mm. Sol. $CHCl_3$. Spar. sol. Et_2O. D_4^{20} 1·2340. Heat of comb. C_v 373·1 Cal.

Leffler, Adams, *J. Am. Chem. Soc.*, 1936, **58**, 1551.
Jeffery, Vogel, *J. Chem. Soc.*, 1934, 1103.
Shriner, Struck, *Organic Syntheses*, 1932, XII, 66.
Jaeger, Fiedler, U.S.P., 1,929,381, (*Chem. Abstracts*, 1934, **28**, 180).

Succinic Dialdehyde.
See Succindialdehyde.

Succinic Semialdehyde.
See 2-Aldehydopropionic Acid.

Succinimide (2 : 5-*Diketopyrrolidine*)



$C_4H_5O_2N$ MW, 99

Plates + $1H_2O$ from EtOH. M.p. 126–7° (125–6°). B.p. 287–8° decomp. Spar. sol. Et_2O. $k = 3 \times 10^{-11}$ at 25°. Forms *N*-metallic derivs.
Oxime: m.p. 197° decomp. *Benzoyl deriv.*: m.p. 184°.
Dioxime: m.p. 207°. *Diacetyl deriv.*: m.p. 170–1°. *Dibenzoyl deriv.*: m.p. 187°.
N-*Acetyl*: b.p. 167°/9·5 mm.
N-*Benzoyl*: m.p. 129–30°.
N-*Me*: *see* *N*-Methylsuccinimide.
N-*Et*: $C_6H_9O_2N$. MW, 127. M.p. 26°. B.p. 236°.
N-2-*Bromoethyl*: $C_6H_8O_2NBr$. 206. M.p. 56–7°.
N-*Propyl*: $C_7H_{11}O_2N$. MW, 141. M.p. 15–16°. B.p. 247–8°/763 mm., 136–7°/27 mm.
N-3-*Bromopropyl*: $C_7H_{10}O_2NBr$. MW, 220. M.p. 52°.
N-*Isopropyl*: m.p. 61°. B.p. 230°/755 mm., 225°/743 mm.
N-sec.-n.-*Butyl*: $C_8H_{13}O_2N$. MW, 155. B.p. 339–40°/758 mm.
N-*Isobutyl*: m.p. 28°. B.p. 247–8°/758 mm.
N-*Isoamyl*: $C_9H_{15}O_2N$. MW, 169, B.p. 261–2°.
N-*Allyl*: $C_7H_9O_2N$. MW, 139. B.p. 249–50°, 244–5°/730 mm., 130–1°/14 mm.
N-*Phenyl*: *see* Succinanil.
N-o-*Nitrophenyl*: $C_{10}H_8O_4N_2$. MW, 220. M.p. 156°.
N-m-*Nitrophenyl*: m.p. 175–6° (172°).
N-p-*Nitrophenyl*: m.p. 208° (203–4°).
N-p-*Hydroxyphenyl*: $C_{10}H_9O_3N$. MW, 191. M.p. 275–6°. *Me ether*: $C_{11}H_{11}O_3N$. MW, 205. M.p. 165°. *Et ether*: pyrantin. $C_{12}H_{13}O_3N$. MW, 219. M.p. 158° (155°). *Propyl ether*: $C_{13}H_{15}O_3N$. MW, 233. M.p. 178°. N-*Benzoyl*: m.p. 215°.
N-o-*Tolyl*: $C_{11}H_{11}O_2N$. MW, 189. M.p. 75°. (101–2°). B.p. 338–40°/733 mm. (339–40°/756 mm.).
N-m-*Tolyl*: m.p. 111–12°. B.p. 340–4°.
N-p-*Tolyl*: m.p. 151°. B.p. 344–5°/733 mm., 212°/13 mm.
N-1-*Naphthyl*: $C_{14}H_{11}O_2N$. MW, 225. M.p. 153°.
N-2-*Naphthyl*: m.p. 183°.
N-*Benzyl*: $C_{11}H_{11}O_2N$. MW, 189. M.p. 98–9° (103°).
N-o-*Nitrobenzyl*: $C_{11}H_{10}O_4N$. MW, 220. M.p. 130°.
N-p-*Nitrobenzyl*: m.p. 150–2°.
N-*Chloro*: cryst. from CCl_4. M.p. 150°.
N-*Bromo*: cryst. from C_6H_6. M.p. 173·5° decomp.

Roeder, *Ber.*, 1913, **46**, 2563.
Ma, Sah, *Chem. Abstracts*, 1934, **28**, 6108.
Fehling, *Ann.*, 1844, **49**, 198.
Tscherniac, *Ber.*, 1901, **34**, 4213.
Swarts, *Am. Chem. J.*, 1897, **19**, 297.

Succinodiformic Acid.
See 1 : 4-Diketoadipic Acid.

Succinonitrile (*Ethylene dicyanide, succinic acid dinitrile,* sym.-*dicyanoethane*)

$CH_2{\cdot}CN$
$CH_2{\cdot}CN$

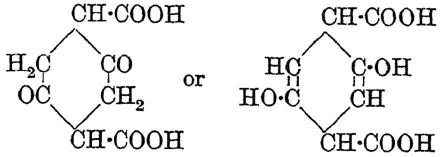
$C_4H_4N_2$ MW, 80

F.p. 53°. M.p. 53·7° (54·5°). B.p. 265–7°, 185°/60 mm., 158–60°/20 mm. Sol. H_2O, EtOH, $CHCl_3$. Spar. sol. Et_2O, CS_2.

Fauconnier, *Bull. soc. chim.*, 1888, **50**, 214.

Succinophenone.
See Diphenacyl.

Succinosuccinic Acid (2 : 5-*Diketocyclohexane*-1 : 4-*dicarboxylic acid,* 2 : 5-*diketohexahydroterephthalic acid, succinylsuccinic acid,* 1 : 4-*dihydroxy*-$\Delta^{1,4}$-*cyclohexadiene*-3 : 6-*dicarboxylic acid*).



I or II

$C_8H_8O_6$ MW, 200

Needles. Spar. sol. H_2O. $FeCl_3 \longrightarrow$ violet col. in EtOH.

Di-Me ester: $C_{10}H_{12}O_6$. MW, 228. Needles from EtOH. M.p. 153°.

Et ester: $C_{10}H_{12}O_6$. MW, 228. M.p. 98° decomp.

Di-Et ester: $C_{12}H_{16}O_6$. MW, 256. (I). M.p. 123°. (II). M.p. 127°.

Propyl ester: $C_{11}H_{14}O_6$. MW, 242. Yellow needles from C_6H_6. M.p. 115° decomp.

Dipropyl ester: $C_{14}H_{20}O_6$. MW, 284. M.p. 91°.

Isobutyl ester: $C_{12}H_{16}O_6$. MW, 256. M.p. 126° decomp.

Di-isobutyl ester: $C_{16}H_{24}O_6$. MW, 312. M.p. 100°.

Diallyl ester: $C_{14}H_{16}O_6$. MW, 280. M.p. 115°.

Liebermann, *Ann.*, 1914, **404**, 287.
Baeyer, Noyes, *Ber.*, 1889, **22**, 2168.
Hantzsch, *Ber.*, 1915, **48**, 772.

Succinperoxide (*Succinic acid peroxide*)

$(HOOC{\cdot}CH_2{\cdot}CH_2{\cdot}CO)_2O_2$

$C_8H_{10}O_8$ MW, 234

Plates. M.p. 128° decomp. Sol. H_2O, EtOH, Me_2CO. Spar. sol. Et_2O. Insol. $CHCl_3$, C_6H_6, ligroin.

Clover, Houghton, *Am. Chem. J.*, 1904, **32**, 55.
Stearns, D.R.P., 170,727, (*Chem. Zentr.*, 1906, II, 79).

Succinyl bromide

$CH_2{\cdot}COBr$
$CH_2{\cdot}COBr$

$C_4H_4O_2Br_2$ MW, 244

B.p. 105–6°/13 mm.

Hughes, Watson, *J. Chem. Soc.*, 1930, 1735.

Succinyl chloride

$CH_2{\cdot}COCl$
$CH_2{\cdot}COCl$

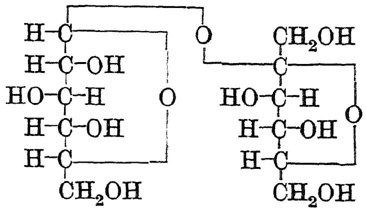
$C_4H_4O_2Cl_2$ MW, 155

Leaflets. M.p. 20° (17°). B.p. 190–2° (193·3°), 150–2°/214 mm., 103–4°/25 mm., 88·5°/19 mm. $D_4^{15\cdot2}$ 1·3948. $n_D^{15\cdot2}$ 1·473.

Vorländer, *Ann.*, 1894, **280**, 183.
Morrell, *J. Chem. Soc.*, 1914, **105**, 1733.
Monsanto, B.P. 418,162, (*Chem. Abstracts*, 1935, **29**, 1436).

Sucrol.
See Dulcin.

Sucrose (*Saccharose, cane sugar*)



$C_{12}H_{22}O_{11}$ MW, 342

Occurs in ripe fruits, sugar cane, beet, etc. Cryst. M.p. 184–5° from dil. EtOH, 179–80° (174°) from EtOH, 169–70° from MeOH. Sweet taste. 100 parts H_2O dissolve 198·6 parts at 12·5°, 245·0 parts at 45°. Sol. MeOH. Spar. sol. 95% EtOH. Insol. Et_2O. $[\alpha]_D^{20}$ + 66·37° in H_2O. $D^{17\cdot5}$ 1·5805, D^{30} 1·5737 from MeOH, 1·5840 from EtOH. Does not reduce Fehling's. Does not form an osazone. Hyd. by dil. acids or by invertase $\longrightarrow$ glucose + fructose. Mod. stable to alkalis. Fermentable. $KMnO_4 \longrightarrow$ oxalic acid + CO_2. $HNO_3 \longrightarrow$ saccharic, tartaric, and oxalic acids. Heat of comb. C_v = 3945·7 Cal. Forms compounds with CaO, BaO and SrO and with halides of alkali metals.

Octa-Me ether: syrup. B.p. 176°/0·05 mm. $[\alpha]_D$ + 69·3° in MeOH, + 66·8° in Me_2CO. D_4^{20} 1·1406. n_D 1·4588. Hyd. $\longrightarrow$ 2 : 3 : 4 : 6-tetramethylglucose + 1 : 3 : 4 : 6-tetramethyl-γ-fructose.

Octapropyl ether: m.p. 45·4–45·5°.

Octanitrate: needles. M.p. 85·5°. Decomp. at 135°. $[\alpha]_D^{20}$ + 55·9° in MeOH. Sol. MeOH, Et_2O. Spar. sol. EtOH, C_6H_6. Insol. H_2O, pet. ether.

Octa-acetyl: needles from EtOH. M.p. 72·3° (69°, 70°). $[\alpha]_D^{20}$ + 59·6° in $CHCl_3$. Sol. hot

EtOH, Et_2O, $CHCl_3$, C_6H_6. Prac. insol. hot H_2O. Insol. pet. ether.

Hexabenzoyl: cryst. M.p. 109°.

Heptabenzoyl: cryst. M.p. 98°.

Avery, Haworth, Hirst, *J. Chem. Soc.*, 1927, 2308.

Josephson, *Ann.*, 1929, **472**, 237.

Hassid, Doudoroff, Barker, *J. Am. Chem. Soc.*, 1944, **66**, 1416.

Hassid, Doudoroff, Barker, Dore, *J. Am. Chem. Soc.*, 1946, **68**, 1465.

Sukesyl Alcohol

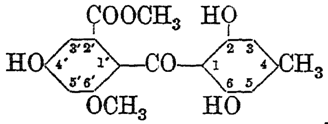
$C_{17}H_{36}O_3$ MW, 288

Obtained from non-saponifiable matter of Sukeso-liver oil. M.p. 64–5°.

Di-phenylurethane: m.p. 79°.

Nakamiya, *Chem. Abstracts*, 1939, **33**, 8176.

Sulochrin



$C_{17}H_{16}O_7$ MW, 332

Mycelial constituent of *Oospora sulphurea-ochracea*. M.p. 262°.

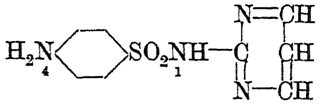
2 : 4'-*Di-Me ether*: m.p. 158°.

Tri-Me ether: plates. M.p. 157°.

Triacetyl: m.p. 164°.

Nishikawa, *Acta Phytochim.*, 1939, **11**, 167; *Bulletin of the Agricultural Chemical Society Japan*, 1940, **16**, 97.

Sulphadiazine (*Sulphapyrimidine, sulphanilamidopyrimidine, N_1-2-pyrimidylsulphanilamide*)



$C_{10}H_{10}O_2N_4S$ MW, 250

Cryst. from H_2O. M.p. 255–6° corr. decomp. Sol. H_2O to 12·3 mgm. per c.c. at 37°. Sol. acids and alkalis. Na deriv. sol. H_2O (sol. p_H 9·6). Used in medicine as bactericide of the sulphanilamide class.

N_4-*Acetyl*: m.p. 258–9° corr. Sol. H_2O to 15 mgm. per c.c. at 37°.

Roblin, Williams, Winnek, English, *J. Am. Chem. Soc.*, 1940, **62**, 2002.

Roblin, Winnek, English, *J. Am. Chem. Soc.* 1942, **64**, 568.

Hartmann, Meyenburg, U.S.P. 2,435,002, (*Chem, Abstracts*, 1948, **42**, 4203).

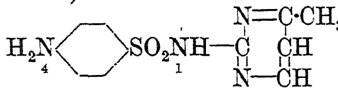
Sulphadimethylpyrimidine.

See Sulphamethazine.

Sulphaguanidine.

See Sulphanilylguanidine.

Sulphamerazine (*2-Sulphanilamido-4-methylpyrimidine, N_1-2-[4-methylpyrimidyl]-sulphanilamide*)



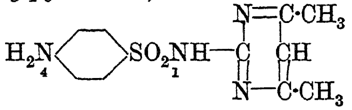
$C_{11}H_{12}O_2N_4S$ MW, 264

M.p. 232–4°. Bactericide of the sulphanilamide class.

N_4-*Acetyl*: m.p. 240°.

Veldstra, Wiardi, *Rec. trav. chim.*, 1942, **61**, 627.

Sulphamethazine (*2-Sulphanilamido-4 : 6-dimethylpyrimidine, N_1-2-[4 : 6-dimethylpyrimidyl]-sulphanilamide, Sulphamezathine, Sulphadimethylpyrimidine*)



$C_{12}H_{14}O_2N_4S$ MW, 278

Pale yellow cryst. + $\frac{1}{2}H_2O$. M.p. 198–9° corr. Sol. H_2O to 75 mgm. per c.c. at 37°. Sol. acids and alkalis. Used in medicine as bactericide of the sulphanilamide class.

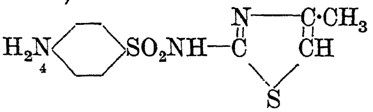
N_4-*Acetyl*: m.p. 249–50° corr.

Roblin, Winnek, English, *J. Am. Chem. Soc.*, 1942, **64**, 568.

Caldwell, Kornfeld, Donnell, *J. Am. Chem. Soc.*, 1941, **63**, 2188.

Rose, Swain, *J. Chem. Soc.*, 1945, 689.

Sulphamethylthiazole (*2-Sulphanilamido-4-methylthiazole*)



$C_{10}H_{11}O_2N_3S_2$ MW, 269

Cryst. from dil. EtOH. M.p. 236–8°. Sol. alkalis. Spar. sol. EtOH. Insol. Et_2O. Bactericide of the sulphanilamide class.

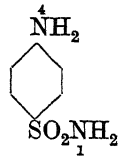
N_4-*Acetyl*: m.p. 259–60°.

Fosbinder, Walter, *J. Am. Chem. Soc.*, 1939, **61**, 2032.

Sulphamezathine.

See Sulphamethazine.

Sulphanilamide (*p-Aminobenzenesulphonamide, Prontosil album*)



$C_6H_8O_2N_2S$ MW, 172

Leaflets from EtOH.Aq. M.p. 165–6° (163°). Sol. MeOH, EtOH, Et_2O, Me_2CO, dil. HCl, hot H_2O. Sol. 632 parts H_2O at 0°, 240 parts at

15°, 47·4 parts at 100°. Spar. sol. pet. ether, $CHCl_3$. Boil with water, acids or alkalis ⟶ sulphanilic acid. Can be diazotised. Has been extensively employed in medicine in the treatment of streptococcal infections, of meningococcal meningitis, etc., and in the treatment of open wounds to prevent gas gangrene.

N_1-*Acetyl*: prisms from H_2O. M.p. 182–4°.

N_4-*Acetyl*: needles from EtOH.Aq. M.p. 219°.

N_1 : N_4-*Diacetyl*: prisms from EtOH. M.p. 253·5–255°.

N_4-*Benzoyl*: cryst. from Py. M.p. 284°.

N_1 : N_4-*Dibenzoyl*: cryst. from EtOH. M.p. 268–70°.

Carranza, Márques, *Chem. Abstracts*, 1940, **34**, 5422.
Gelmo, *J. prakt. Chem.*, 1908, **77**, 372.
Northey, *Chem. Reviews*, 1940, **27**, 85 (*Bibl.*).
Dewing, Gray, Platt, Stephenson, *J. Chem. Soc.*, 1942, 239.

Sulphanilamidodimethylpyrimidine.
See Sulphamethazine.

Sulphanilamidomethylpyrimidine.
See Sulphamerazine.

Sulphanilamidomethylthiazole.
See Sulphamethylthiazole.

Sulphanilamidopyrazine.
See Sulphapyrazine.

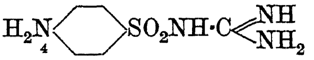
Sulphanilamidopyridine.
See Sulphapyridine.

Sulphanilamidopyrimidine.
See Sulphadiazine.

Sulphanilamidothiazole.
See Sulphathiazole.

Sulphanilic Acid.
See Aniline-*p*-sulphonic Acid.

Sulphanilylguanidine (*Sulphaguanidine*)



$C_7H_{10}O_2N_4S$ MW, 214

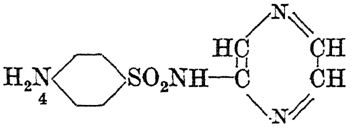
Cryst. + $1H_2O$ from H_2O. M.p. 142·5–143·5° (sealed tube). M.p. anhyd. 189–90°. Sol. to 10% in boiling H_2O, 5% in boiling 95% EtOH, 1·5% in boiling acetone. Spar. sol. cold H_2O, cold EtOH. Sol. dil. min. acids; insol. dil. aq. alkalis. Insol. Et_2O, C_6H_6. Used in medicine as bactericide particularly for bacillary dysentery.

Hydrochloride: m.p. 205–6°.

N_4-*Acetyl*: m.p. 262–6° (248–51°).

Marshall *et al.*, *Bulletin of the Johns Hopkins Hospital*, 1940, **67**, 163, (*Chem. Abstracts*, 1940, **34**, 7405).
Dewing, Smith, *Nature*, 1941, **148**, 24.
Birtwell, Haworth, Rose, Swain, Vasey, *J. Chem. Soc.*, 1946, 491.
Winnek, Faith, U.S.P. 2,380,006, (*Chem. Abstracts*, 1945, **39**, 5410).

Sulphapyrazine (*2-Sulphanilamidopyrazine*)



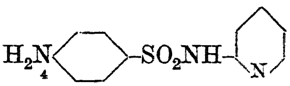
$C_{10}H_{10}O_2N_4S$ MW, 250

Needles from $PhNO_2$. M.p. 251°. Sol. Py, quinoline, isoquinoline, boiling $PhNO_2$. Insol. H_2O, EtOH, Et_2O, dioxan, AcOEt, $CHCl_3$, C_6H_6.

N_4-*Acetyl*: m.p. 240–2° (255–7°).

Sausville, Spoerri, *J. Am. Chem. Soc.*, 1941, **63**, 3153.

Sulphapyridine (p-*Amino*-N-*2-pyridylbenzenesulphonamide*, *2-sulphanilamidopyridine*, *M. & B.* 693, *Dagenan*, ω-N-*2-pyridylsulphanilamide*)



$C_{11}H_{11}O_2N_3S$ MW, 249

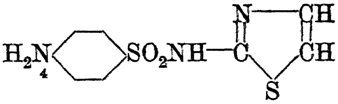
Cryst. from EtOH. M.p. 190–1° (191–2°). Sol. H_2O to 49·5 mgm. per 100 c.c. at 37°. Extensively employed in medicine in the treatment of bacterial infections, especially pneumonia.

N_4-*Acetyl*: needles from Me_2CO. M.p. 226–7° (225°).

Márques, *Chem. Abstracts*, 1940, **34**, 5422.
Winterbottom, *J. Am. Chem. Soc.*, 1940, **62**, 160.
Roblin, Winnek, *J. Am. Chem. Soc.*, *ibid.*, 1999.
Shen, Chen, *Chem. Abstracts*, 1943, **37**, 352.
Pearl, *J. Org. Chem.*, 1945, **10**, 205.

Sulphapyrimidine.
See Sulphadiazine.

Sulphathiazole (*2-Sulphanilamidothiazole*)



$C_9H_9O_2N_3S_2$ MW, 255

Cryst. powder from 45% EtOH. M.p. 202–202·5°. Sol. H_2O to 60 mgm. per 100 c.c. at 26° (p_H 6·03), 94 mgm. at 37°. Sol. EtOH to 525 mgm. per 100 c.c. at 26°. Used in medicine as a bactericide particularly in treatment of streptococcal and staphylococcal infections, pneumonia and gonorrhea.

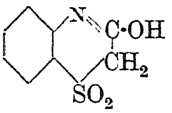
Hydrochloride: m.p. 193–7°.

Na deriv.: m.p. 264·5–265°. pH of 2% aq. sol. 9·57.

N_4-*Acetyl*: m.p. 256–7°.

Lott, Bergheim, *J. Am. Chem. Soc.*, 1939, **61**, 3592.
Fosbinder, Walter, *ibid.*, 2032.
Delivala, Ganapathi, Shirsat, *Chem. Abstracts*, 1944, **38**, 4573.

Sulphazone



$C_8H_7O_3NS$ MW, 197

Brown leaflets from EtOH.Aq. M.p. 207–8°. Sol. EtOH, Me_2CO, AcOH. Spar. sol. Et_2O, hot H_2O. Insol. C_6H_6, ligroin.

B,HNO$_3$: yellow leaflets. Explodes at 172°.

Me ether: $C_9H_9O_3NS$. MW, 211. Leaflets from MeOH.Aq. M.p. 210°.

Claasz, *Ber.*, 1916, **49**, 614.
M.L.B., D.R.P., 269,428, (*Chem. Abstracts*, 1914, **8**, 2035).

Sulphido-α-alanine.
See Lanthionine.

Sulphoacetic Acid

$HO_3S{\cdot}CH_2{\cdot}COOH$

$C_2H_4O_5S$ MW, 140

Hygroscopic cryst. + $1H_2O$ from H_2O. M.p. 84–6° (about 75°). Mod. sol. EtOH, Me_2CO. Insol. Et_2O, $CHCl_3$.

C-*Anilide*: *Na salt*, m.p. 284°. *Aniline salt*, m.p. 229–31°.

C-o-*Toluidide*: *Na salt*, m.p. 223–4°. o-*Toluidine salt*: m.p. 189–91°.

C-p-*Anisidide*: p-*anisidine salt*, m.p. 224–7°.

C-p-*Phenetidide*: *Na salt*, m.p. 270°. p-*Phenetidine salt*: m.p. 233–40°.

C-1-*Naphthalide*: *Na salt*, m.p. 285°.

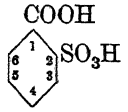
Stillich, *J. prakt. Chem.*, 1906, **73**, 538; 1906, **74**, 53.
Fichter, *Z. Elektrochem*, 1914, **20**, 471.
Lichtenhahn, *Ber.*, 1915, **48**, 1950.

Sulphoalanine.
See Cysteic Acid *and* Isocysteic Acid.

Sulphoanisic Acid.
See under 4-Hydroxysulphobenzoic Acid.

Sulphobenzide.
See Diphenyl sulphone.

o-Sulphobenzoic Acid (*Benzoic acid* o-*sulphonic acid*)



$C_7H_6O_5S$ MW, 202

Needles + $3H_2O$ from H_2O. M.p. 68–9°, anhyd. 134° (141°). Sol. H_2O, EtOH. Insol. Et_2O. Heat ⟶ anhydride.

Di-benzylthiouronium salt: white needles from EtOH. M.p. 205–6°.

1-*Me ester*: *chloride*. $C_8H_7O_4ClS$. MW, 234·5. M.p. 64–5°. Sol. EtOH, Et_2O. *Amide*: $C_8H_9O_4NS$. MW, 215. M.p. 125–6°.

1-*Et ester*: *amide*. $C_9H_{11}O_4NS$. MW, 229. M.p. 84°.

Di-Et ester: $C_{11}H_{14}O_5S$. MW, 258. B.p. 212–13°/21 mm.

1-*Phenyl ester*: $C_{13}H_{10}O_5S$. MW, 278. Needles from H_2O. M.p. 277–80°. Sol. H_2O, AcOH. Spar. sol. EtOH. *Chloride*: $C_{13}H_9O_4ClS$. MW, 296·5. Prisms from AcOH. M.p. 103–4°. Sol. EtOH, AcOH. Spar. sol. Et_2O, C_6H_6. *Amide*: $C_{13}H_{11}O_4NS$. MW, 277. Prisms. M.p. 132°.

2-*Phenyl ester*: *amide*. M.p. 95°.

Diphenyl ester: $C_{19}H_{14}O_5S$. MW, 354. Needles from AcOH. M.p. 117·5–118·5°.

1-o-*Tolyl ester*: *chloride*. $C_{14}H_{11}O_4ClS$. MW, 310·5. M.p. 112°. *Amide*: $C_{14}H_{13}O_4NS$. MW, 291. M.p. 152°.

2-*Chloride*: *nitrile*. $C_7H_4O_2NClS$. MW, 201·5. Prisms from Et_2O. M.p. 69–70° (67·5°). Sol. Et_2O, $CHCl_3$, C_6H_6, hot pet. ether.

Dichloride: $C_7H_4O_3Cl_2S$. MW, 239. (i) Needles from pet. ether. M.p. 79°. Sol. Et_2O, $CHCl_3$. Spar. sol. pet. ether. (ii) Plates from Et_2O. M.p. 40°. Sol. Et_2O, $CHCl_3$.

1-*Amide*: $C_7H_7O_4NS$. MW, 201. Prisms + $1H_2O$. M.p. anhyd. 185–6°, (193–4°). Sol. H_2O, EtOH.

2-*Amide*: plates from H_2O. M.p. 165–7° (rapid heat.), 153–5° (slow heat.). Sol. H_2O, EtOH, Et_2O. $k = 2{\cdot}06 \times 10^{-3}$ at 25°. *K deriv.*: m.p. 285–6°. o-*Toluidide*: m.p. 193°. p-*Toluidide*: m.p. 202°. *Hydrazide*: m.p. 182°.

1-*Methylamide*: $C_8H_9O_4NS$. MW, 215. M.p. 126°.

1-*Ethylamide*: $C_9H_{11}O_4NS$. MW, 229. M.p. about 111°.

Imide: *see* Saccharin.

Anhydride: $C_7H_4O_4S$. MW, 184. Plates from C_6H_6. M.p. 129·5° (128°). B.p. 184–6°/18 mm. Sol. Et_2O, C_6H_6, $CHCl_3$, hot H_2O.

Nitrile: o-cyanobenzenesulphonic acid. $C_7H_5O_3NS$. MW, 183. Needles from H_2O. M.p. 279–279·5°. Mod. sol. EtOH, hot H_2O. Spar. sol. Et_2O, $CHCl_3$. *Anilide*: m.p. 150–2°.

2-*Anilide*: m.p. 156°.

Dianilide: m.p. 194–5°.

Maarse, *Rec. trav. chim.*, 1914, **33**, 210.
Krannich, *Ber.*, 1900, **33**, 3485.
White, Acree, *Organic Syntheses*, Collective Vol. I, 13, 482.
Auger, Vary, *Bull. soc. chim.*, 1921, **29**, 990.
Ray, Dey, *J. Chem. Soc.*, 1920, **117**, 1406.
Campaigne, Suter, *J. Am. Chem. Soc.*, 1942, **64**, 3040.

m-Sulphobenzoic Acid (*Benzoic acid* m-*sulphonic acid*).

Cryst. + $2H_2O$. M.p. 98°, anhyd. 141°. Sol. H_2O, EtOH. Insol. C_6H_6, $CHCl_3$, pet. ether. Anhyd. acid sol. Et_2O.

1-*Me ester*: $C_8H_8O_5S$. MW, 216. M.p. 65°. *Chloride*: $C_8H_7O_4ClS$. MW, 234·5. M.p. 63–5°.

3-*Me ester*: m.p. 139–40°.

Di-Me ester: $C_9H_{10}O_5S$. MW, 230. M.p. 32–3°. B.p. 198–200°/20 mm.

3-*Chloride*: $C_7H_5O_4ClS$. MW, 220·5. M.p. 133–4°.

Dichloride: $C_7H_4O_3Cl_2S$. MW, 239. M.p. 20·4°.

3-*Amide*: $C_7H_7O_4NS$. MW, 201. M.p. 237–8° (233°). *Nitrile*: $C_7H_6O_2N_2S$. MW, 182. M.p. 151–2°.

Diamide: $C_7H_8O_3N_2S$. MW, 200. M.p. anhyd. 170°.

Offermann, *Ann.*, 1894, **280**, 6.
Maarse, *Rec. trav. chim.*, 1914, **33**, 209.
Nakaseko, *Am. Chem. J.*, 1912, **47**, 448.

p-Sulphobenzoic Acid (*Benzoic acid* p-*sulphonic acid*).

Needles + $3H_2O$. M.p. 94°, anhyd. 259–60°. Sol. H_2O, EtOH. Anhyd. acid sol. Et_2O.

Benzylthiouronium salt: plates from EtOH. M.p. 212–14°.

1-*Me ester*: m.p. 99–100°.

4-*Me ester*: m.p. 195–6°.

Di-Me ester: m.p. 88–90°.

1-*Et ester*: *amide*. $C_9H_{11}O_4NS$. MW, 229. M.p. 110–11° ⟶ solid, m.p. 94–5°.

4-*Chloride*: *nitrile*. $C_7H_4O_2NClS$. MW, 201·5. M.p. 111–12°.

Dichloride: m.p. 56–7°.

4-*Amide*: decomp. at 280°. $k = 2{\cdot}56 \times 10^{-4}$ at 25°. N-*Dichloro*: *see* Halazone. *Nitrile*: $C_7H_6O_2N_2S$. MW, 182. M.p. 168–9°.

Diamide: m.p. 236°.

Nitrile: *anilide*. M.p. 112°. m-*Toluidide*: m.p. 128°.

Dianilide: m.p. 252–3° decomp.

Di-m-*toluidide*: m.p. 241–2° decomp.

Heinemann, B.P. 23,575, (*Chem. Abstracts*, 1916, **10**, 1579).
Remsen, *Ann.*, 1875, **178**, 288.
Maarse, *Rec. trav. chim.*, 1914, **33**, 209.
Campaigne, Suter, *J. Am. Chem. Soc.*, 1942, **64**, 3040.

Sulphocresotic Acid.

See Hydroxy-sulphotoluic Acid.

Sulphoethylurea.

See Taurocarbamic Acid.

2-Sulphoethyl Alcohol.

See Isethionic Acid.

Sulphonal (*Acetone diethylsulphone, propane-2 : 2-diethyldisulphone*)

$C_7H_{16}O_4S_2$ MW, 228

Prisms from EtOH, microscopic leaflets from Et_2O. M.p. 125·8°. B.p. 300° (slight decomp.). Sublimes (begins at 66°). Volatile in steam. Solubilities (1 gm. in x gms. solvent): H_2O, 422 at 18°, 222 at 37°, 8 at 100°; EtOH, 2 at 78°; Et_2O, 67 at 19°, 79 at 15°; $CHCl_3$, 3·3 at 20°; C_6H_6, 12·7 at 20°; AcOEt, 13·7 at 20°; CCl_4, 110 at 20°. Stable to acids, alkalis, bromine. Hypnotic.

Falck, *Chem. Abstracts*, 1920, **14**, 1002.
Hirayama, Matsuzaki, Okamoto, *Chem. Abstracts*, 1918, **12**, 1586.

Sulphonyldiacetic Acid (*Dimethylsulphone-α : α′-dicarboxylic acid*)

$HOOC{\cdot}CH_2{\cdot}SO_2{\cdot}CH_2{\cdot}COOH$

$C_4H_6O_6S$ MW, 182

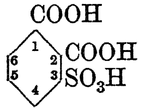
Plates. M.p. 182°. Decomp. at 200° ⟶ dimethyl sulphone. Very sol. H_2O, EtOH. Mod. sol. H_2SO_4.Aq., Et_2O. k (first) $= 1{\cdot}3 \times 10^{-2}$; (second) $= 4{\cdot}75 \times 10^{-4}$ at 25°. Electrolysis of K salts ⟶ $H_2SO_4 + CO_2$. $NaNO_2$.Aq. ⟶ $CO_2 + HCN + H_2SO_4$.

Fichter, Krummenacher, *Helv. Chim. Acta*, 1918, **1**, 162.
Lovén, *Ber.*, 1884, **17**, 2818.

Sulphonyldiphenylmethane.

See Diphenylmethane sulphone.

3-Sulphophthalic Acid (*Phthalic acid* 3-*sulphonic acid*)



$C_8H_6O_7S$ MW, 246

Prisms + H_2O of cryst. M.p. 62–4°. Very sol. H_2O, EtOH. Insol. Et_2O.

3-*Amide*: $C_8H_7O_6NS$. MW, 245. Needles + $1H_2O$ from H_2O. M.p. 155° decomp. (softens at 120°). *Di-Me ester*: m.p. 135°. *Di-Et ester*: m.p. 102°.

Remsen, Stokes, *Am. Chem. J.*, 1884, **6**, 279.
Moulton, *Am. Chem. J.*, 1891, **13**, 203.
Cf. Rée, *Ann.*, 1886, **233**, 217.

4-Sulphophthalic Acid (*Phthalic acid* 4-*sulphonic acid*).

Cryst. + $1H_2O$. M.p. 138–40°. Very sol. H_2O, EtOH. Insol. Et_2O. Fused with NaOH at 220° ⟶ 4-hydroxyphthalic acid. K salt heated with Na formate ⟶ trimellitic acid.

4-*Chloride*: $C_8H_5O_6ClS$. MW, 264·5. Prisms from Et_2O. M.p. 167–70° decomp.

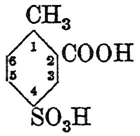
4-*Amide*: prisms from H_2O. M.p. 192–200° decomp.

Bentley, Weizmann, *J. Chem. Soc.*, 1907, **91**, 100.
Rée, *Ann.*, 1886, **233**, 219, 228.

Sulphosalicylic Acid.

See 2-Hydroxy-4-sulphobenzoic Acid *and* 6-Hydroxy-3-sulphobenzoic Acid.

4-Sulpho-*o*-toluic Acid



$C_8H_8O_5S$ MW, 216

Fibrous cryst. mass. Very sol. H_2O. Fused with KOH ⟶ 4-hydroxy-*o*-toluic acid.

4-*Amide*: $C_8H_9O_4NS$. MW, 215. Needles. M.p. 243°.

Jacobsen, Wierss, *Ber.*, 1883, **16**, 1959.
Baudisch, Perkin, *J. Chem. Soc.*, 1909, **95**, 1883.

5-Sulpho-*o*-toluic Acid.

Mono-NH_4 salt: prisms from H_2O. M.p. 284°. Reacts acid.

2-*Amide*: $C_8H_9O_4NS$. MW, 215. Needles from H_2O. M.p. 217°. *NH_4 salt*: prisms. M.p. 276–8°.

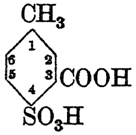
5-*Amide*: m.p. 211°.

Amide-nitrile: $C_8H_8O_2N_2S$. MW, 196. Cryst. M.p. 160°.

Chloride-nitrile: $C_8H_6O_2NClS$. MW, 215·5. Cryst. from ligroin. M.p. 53°.

Nowell, *Am. Chem. J.*, 1912, **48**, 225, 237.

4-Sulpho-*m*-toluic Acid.



$C_8H_8O_5S$ MW, 216

Fused with KOH ⟶ 4-hydroxy-*m*-toluic acid.

Meldrum, Perkin, *J. Chem. Soc.*, 1909, **95**, 1891.

5-Sulpho-*m*-toluic Acid.

Cryst. + $2H_2O$ from H_2O. M.p. 110°. Fused with KOH ⟶ 5-hydroxy-*m*-toluic acid.

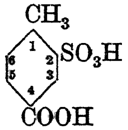
Meldrum, Perkin, *J. Chem. Soc.*, 1909, **95**, 1891.

6-Sulpho-*m*-toluic Acid.

6-*Amide*: needles from H_2O. M.p. 254° (220° slow heat.).

Waters, *Am. Chem. J.*, 1912, **47**, 343, 349,
Meldrum, Perkin, *J. Chem. Soc.*, 1909. **95**, 1891.

2-Sulpho-*p*-toluic Acid



$C_8H_8O_5S$ MW, 216

Cryst. + $1H_2O$ from AcOH, + $3H_2O$ from H_2O.

2-*Amide*: needles from H_2O. M.p. 267°.

Diamide: $C_8H_{10}O_3N_2S$. MW, 214. Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. 228°.

Meldrum, Perkin, *J. Chem. Soc.*, 1908, **93**, 1419.
Weinreich, *Ber.*, 1887, **20**, 981.

3-Sulpho-*p*-toluic Acid.

Cryst. + $3H_2O$. M.p. anhyd. 190° (158°). Sol. H_2O. Spar. sol. Et_2O, $CHCl_3$, C_6H_6. Fused with KOH ⟶ *m*-cresotic acid.

3-*Amide*: cryst. from H_2O. M.p. 185° (181°). *Me ester*: m.p. 145°. *Et ester*: m.p. 95°.

4-*Amide*: *NH_4 salt*, cryst. + $1H_2O$. M.p. 186°.

Amide-nitrile: leaflets from Py. Very spar. sol. H_2O, Et_2O. Sol. alkalis.

Dichloride: $C_8H_6O_3Cl_2S$. MW, 253. Cryst. from pet. ether. M.p. 59°.

Chloride-nitrile: leaflets from ligroin. M.p. 67°.

Weber, *Ber.*, 1892, **25**, 1741.
Randall, *Am. Chem. J.*, 1891, **13**, 258.

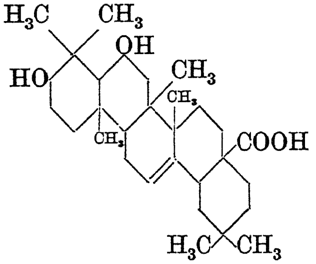
Sulphovinic Acid.

See Ethyl hydrogen sulphate.

Sulphuric Ether.

See Diethyl Ether.

Sumaresinolic Acid



$C_{31}H_{50}O_4$ MW, 486

Isolated from Sumatra benzoin. Needles from EtOH.Aq. M.p. 298–9°. $[\alpha]_D^{24}$ + 102·2° in $CHCl_3$.

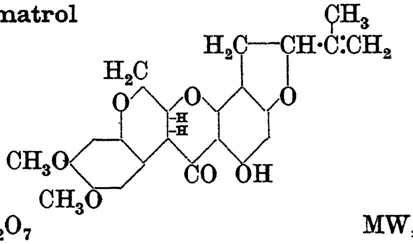
Me ester: cryst. from MeOH.Aq. M.p. 220–1°. $[\alpha]_D^{24}$ + 53·6° in $CHCl_3$.

Et ester: needles from EtOH.Aq. M.p. 207–8°.

Bromolactone: needles. M.p. 252°. $[\alpha]_D^{24}$ + 60·2° in $CHCl_3$.

Winterstein, Egli, *Z. physiol. Chem.*, 1931, **202**, 207.
Zinke, Liebe, *Monatsh.*, 1918, **39**, 219.
Ruzicka, Jeger, Grob, Hösli, *Helv. Chim. Acta*, 1943, **26**, 2283.
Ruzicka, Norymberski, Jeger, *Helv. Chim. Acta*, 1945, **28**, 380.

Sumatrol



$C_{23}H_{22}O_7$ MW, 410

Occurs in resin from *Derris* species. Needles from EtOH. M.p. 195–6° (air-dried). Recryst. from Me_2CO, m.p. 183° ⟶ 194° after several days keeping. Sol. $CHCl_3$. Mod. sol. C_6H_6, AcOEt. Spar. sol. MeOH, cold AcOH, 8% NaOH.Aq. $[\alpha]_D - 184°$ in C_6H_6. Alc. $FeCl_3$ ⟶ deep brownish-green col.

Acetyl: m.p. 218–19°. $[\alpha]_D + 57·6°$ in C_6H_6, − 20·5° in Me_2CO.

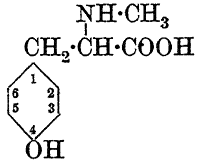
Oxime: needles from EtOH.Aq. M.p. 245–7°.

Robertson, Rusby, *J. Chem. Soc.*, 1937, 497.
Cahn, Phiper, Boam, *J. Chem. Soc.*, 1938, 513.
Kenny, Robertson, George, *J. Chem. Soc.*, 1939, 1601.

Superpalite.
See Trichloromethyl chloroformate.

Suprarenin.
See Adrenaline.

Surinamine (*Ratanhin, geoffroyin, angelin, andirin*, N-*methyltyrosine*)



$C_{10}H_{13}O_3N$ MW, 195

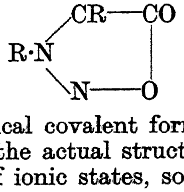
Occurs in bark of *Andira retusa*, H.B. and K. Needles. M.p. 257° (decomp. at 280°). $[\alpha]_D^{21}$ + 19·8° in dil. HCl.

Me ester: $C_{11}H_{15}O_3N$. MW, 209. M.p. 111–12° (116–17°).

Me ether-nitrile: $C_{11}H_{14}ON_2$. MW, 190. M.p. 152–3°.

Fischer, Lipschitz, *Ber.*, 1915, **48**, 377.
Winterstein, *Z. physiol. Chem.*, 1919, **105**, 20.
Kanevska, *J. prakt. Chem.*, 1929, **124**, 48.

Sydnone.
The name given to the unknown parent compound (R = H) of a group of substances which possess the following arrangement of atoms:

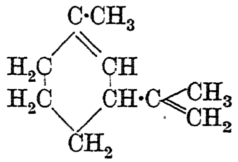


A unique classical covalent formula cannot be presented and the actual structure is a hybrid of a number of ionic states, some dipolar and some tetrapolar.

Eade, Earl, *J. Chem. Soc.*, 1946, 591.
Baker, Ollis, Poole, *J. Chem. Soc.*, 1949, 307.

Sylvan.
See 2-Methylfuran.

Sylvestrene ($\Delta^{1,8(9)}$-m-*Menthadiene*, 1-*methyl*-3-*isopropenylcyclohexene*)



$C_{10}H_{16}$ MW, 136

Terpene hydrocarbon which does not occur in nature, but is generated from *carene* hydrocarbons during processes of isolation and purification of essential oils of *Pinus*. All forms sol. Ac_2O + 1 drop conc. H_2SO_4 ⟶ deep blue col.

d-.
B.p. 175°/751 mm. Polymerises on long heating. D^{18} 0·8479. n_D^{18} 1·4760. $[\alpha]_D^{18} + 83·18°$. Heat of comb. C_v 1464·2 Cal.

Dihydrochloride: m.p. 72°.
Nitrosochloride: m.p. 106–7°.

l-.
B.p. 176–8°. D^{19} 0·848. n_D^{18} 1·4761. $[\alpha]_D^{18}$ − 68·2° in AcOEt.

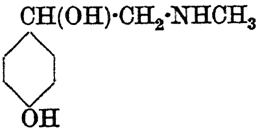
Dihydrochloride: m.p. 72°.

dl-. Carvestrene.
B.p. 178°. Resinifies in air.
Dihydrochloride: cryst. from MeOH. M.p. 52°.

Aschan, *Ann.*, 1928, **461**, 1.
Semmler, Schiller, *Ber.*, 1927, **60**, 1591.
Rao, Simonsen, *J. Chem. Soc.*, 1925, 2494.
Haworth, Perkin, Wallach, *J. Chem. Soc.*, 1913, **103**, 2233.

Sylvic Acid.
See Abietic Acid.

Sympathol (1-[p-*Hydroxyphenyl*]-N-*methylaminoethyl alcohol, synephrine*, p-[α-*hydroxy*-β-*methylaminoethyl*]-*phenol*)



$C_9H_{13}O_2N$ MW, 167

M.p. 184–5° decomp. Spar. sol. org. solvents.

B,HCl: cryst. powder. M.p. 149·5–151°. $FeCl_3$ ⟶ violet col. Phosphotungstic acid ⟶ blue col. Vasoconstrictor. Substitute for epinephrine.

Diacetyl: m.p. 157°.
Dibenzoyl: m.p. 176°.

Priestley, Moness, *J. Org. Chem.*, 1940, **5**, 355.
Gordon, *Chem. Zentr.*, 1931, II, 3016.
Corrigan, Langerman, Moore, *J. Am. Chem. Soc.*, 1945, **67**, 1894.
Feodor, Kovács, *J. Am. Chem. Soc.*, 1949, **71**, 1045.

Synephrine.
See Sympathol.

Synthalin (*Decamethylenediguanidine*, 1 : 10-*diguanidino*-n-*decane*)

$H_2N \cdot C(:NH) \cdot NH \cdot [CH_2]_{10} \cdot NH \cdot C(:NH) \cdot NH_2$

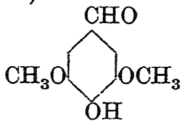
$C_{12}H_{28}N_6$ MW, 256

Antidiabetic.

B,2HCl : cryst. from EtOH–Et_2O. M.p. 193°.

Kumagai *et al.*, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1928, **9**, 271.
Schering-Kahlbaum A.G., B.P. 285,873, (*Chem. Abstracts*, 1929, **23**, 154).
Ammon, *Chem.-Tech. Rundschau*, 1930, **45**, 406 (*Review*).

Syringa-aldehyde (*Syringic aldehyde*, 4-*hydroxy*-3 : 5-*dimethoxybenzaldehyde*, *gallaldehyde* 3 : 5-*dimethyl ether*)



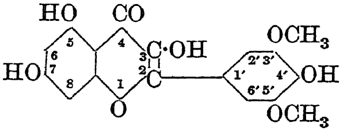
$C_9H_{10}O_4$ MW, 182

M.p. 113°. B.p. 192–3°/14 mm. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, hot C_6H_6. $FeCl_3$.Aq. ⟶ olive-green col. The K and Na salts are yellow.

O-*Carbomethoxyl* : m.p. 98–9°.
Oxime : m.p. 91°.
Hydrazone : m.p. 208–9°.
p-*Nitrophenylhydrazone* : m.p. 216–17°.
Semicarbazone : m.p. 188°.

Sharp, *J. Chem. Soc.*, 1937, 853.
Mauthner, *J. prakt. Chem.*, 1935, **142**, 26.
McCord, *J. Am. Chem. Soc.*, 1931, **53**, 4181.
Pauly, Strassberger, *Ber.*, 1929, **62**, 2277.
Pearl, *J. Am. Chem. Soc.*, 1948, **70**, 1746.

Syringetin (5 : 7 : 4′-*Trihydroxy*-3′ : 5′-*dimethoxyflavonol*)



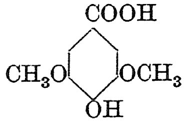
$C_{17}H_{14}O_8$ MW, 346

Pale yellow needles from AcOH. M.p. 288–9° (darkens at 270°). Mod. sol. MeOH, EtOH, Me_2CO, AcOH. Spar. sol. AcOEt, $CHCl_3$. Insol. C_6H_6, pet. ether. Conc. H_2SO_4 ⟶ yellow col. with green fluor. Alk. sols. are intensely deep yellow.

Tetra-acetyl : m.p. 224–6°.
4′-*Benzyl ether* : m.p. 240–1°. *Triacetyl* : m.p. 191–4°.

Heap, Robinson, *J. Chem. Soc.*, 1929, 67.

Syringic Acid (4-*Hydroxy*-3 : 5-*dimethoxybenzoic acid*, *gallic acid* 3 : 5-*dimethyl ether*)



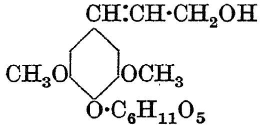
$C_9H_{10}O_5$ MW, 198

Cryst. M.p. 204–5°.
4-*Acetyl* : m.p. 191° (187°).
4-*Benzoyl* : m.p. 229–32°. *Chloride* : m.p. 117–18°. *Anhydride* : m.p. 112–13°.
Me ester : $C_{10}H_{12}O_5$. MW, 212. M.p. 83–4°, anhyd. 107°. 4-*Acetyl* : m.p. 131·5°.
Et ester : $C_{11}H_{14}O_5$. MW, 226. M.p. 56°.
4-*Et ether* : 3 : 5-dimethoxy-4-ethoxybenzoic acid. $C_{11}H_{14}O_5$. MW, 226. M.p. 123–4°. *Me ester* : m.p. 65°. *Et ester* : m.p. 47°. *Amide* : m.p. 155°.

Wassmuth, *Ber.*, 1934, **67**, 701.
Bogert, Coyne, *J. Am. Chem. Soc.*, 1929, **51**, 571.
Heap, Robinson, *J. Chem. Soc.*, 1929, 67.

Syringic Aldehyde.
See Syringa-aldehyde.

Syringin (*Methoxyconiferin*)

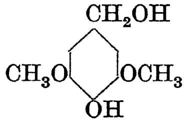


$C_{17}H_{24}O_9$ MW, 372

Glucoside occurring in various species of *Syringa*, *Ligustrum*, and *Jasminum*. Needles + $1H_2O$ from H_2O. M.p. 191–2°. $[\alpha]_D$ − 17·1°. Sol. hot H_2O, EtOH. Very spar. sol. cold H_2O. Insol. Et_2O. Tasteless. Does not reduce Fehling's or Tollen's. No col. with $FeCl_3$. Not pptd. by $Pb(OAc)_2$. Sol. conc. HNO_3 ⟶ blood-red col. Sol. conc. H_2SO_4 ⟶ dark blue to violet col. Hyd. by emulsin.

Pauly, Strassberger, *Ber.*, 1929, **62**, 2277.

Syringyl Alcohol (4-*Hydroxy*-3 : 5-*dimethoxybenzyl alcohol*, *gallyl alcohol* 3 : 5-*dimethyl ether*)



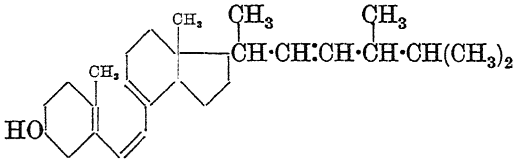
$C_9H_{12}O_4$ MW, 184

Cryst. from C_6H_6, AcOEt. M.p. 136°.

Richtzenhain, *Ber.*, 1944, **77**, 409.

T

Tachysterol



$C_{28}H_{44}O$ MW, 396

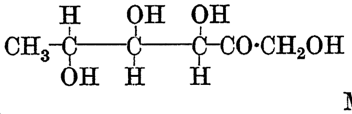
B.p. 220°/high vac. Sol. most org. solvents. $[\alpha]^{18}_{D}$ − 70° (− 11·5°) in C_6H_6. Absorption maximum at 2800 Å. with inflexions at 2940 and 2680 Å. Irradiation ⟶ calciferol (vitamin D_2).

3 : *5-Dinitro-*p*-toluyl* : cryst. from Me_2CO–MeOH or Et_2O–MeOH. M.p. 154·5°. $[\alpha]^{16}_{D}$ + 40·4° in $CHCl_3$.

Citraconic anhydride adduct of acetyl : needles from AcOH.Aq. M.p. 161–2°. $[\alpha]^{20}_{D}$ + 75·2° in $CHCl_3$.

Windaus, Deppe, Wunderlich, *Ann.*, 1938, **533**, 118.
Windaus, Werder, Luttringhaus, Fernholz, *Ann.*, 1932, **499**, 188.
Lettré, *Ann.*, 1934, **511**, 280.
Grundmann, *Z. physiol. Chem.*, 1938, **252**, 151.

Tagatomethylose (*6-Deoxytagatose*)



$C_6H_{12}O_5$ MW, 164

d-.

Colourless syrup. $[\alpha]^{18}_{D}$ − 2 ± 2°. No mutarotation. Reduces Fehling's.

o-*Nitrophenylhydrazone* : m.p. 160–1°. $[\alpha]^{17}_{D}$ + 72°.

l-.

Colourless cryst. from AcOEt. M.p. 68–9°. $[\alpha]^{24\cdot5}_{D}$ + 3·4°. No mutarotation. Astringent taste. Reduces Fehling's.

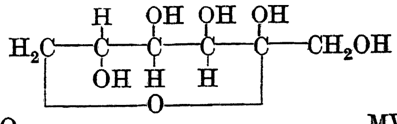
o-*Nitrophenylhydrazone* : orange cryst. M.p. 161–2°. $[\alpha]^{20}_{D}$ − 69°.

dl-.

o-*Nitrophenylhydrazone* : m.p. 162–3°. "$[\alpha]^{16}_{D}$ + 5°."

Barnett, Reichstein, *Helv. Chim. Acta*, 1937, **20**, 1529; 1938, **21**, 913.

Tagatose



$C_6H_{12}O_6$ MW, 180

d-.

Cryst. M.p. 134–5° (124°). $[\alpha]^{22}_{D}$ + 1·0° in H_2O ($[\alpha]_D$ − 2·3° in H_2O). Sol. H_2O. Spar. sol. EtOH. Reduces Fehling's. Mod. sweet taste. Non-fermentable.

Phenylosazone : yellow needles. M.p. 193–4°. Identical with *d*-galactosazone.

Diacetone deriv. : m.p. 65–6°. $[\alpha]^{20}_{D}$ + 81·5° in Me_2CO, + 71·8° in H_2O.

α-Methyltagatoside : m.p. 128°. $[\alpha]_D$ + 56·8°.

Tetra-Me ether : b.p. 40°/10^{-4} mm. $[\alpha]_D$ + 21·4° in MeOH. D^{19}_{4} 1·1087. n^{19}_{D} 1·1446.

Tetra-Me ether : b.p. 52°/10^{-4} mm. $[\alpha]_D$ − 3·4° in MeOH.

α-Penta-acetyl : m.p. 132°. $[\alpha]_D$ + 20·2° in $CHCl_3$, − 25° in MeOH.

l-.

Cryst. M.p. 162°. $[\alpha]_D$ − 3·9° in H_2O.

Phenylosazone : m.p. 183–4°.

dl-. Dulcitose.

NaHg ⟶ *dl*-talitol.

Methylphenylosazone : needles from Py.Aq. M.p. 148–50°.

de Bruyn, v. Ekenstein, *Rec. trav. chim.*, 1897, **16**, 265.
Neuberg, *Ber.*, 1902, **35**, 2629.
Nef, *Ann.*, 1914, **403**, 341.
Reichstein, Bosshard, *Helv. Chim. Acta*, 1934, **17**, 753.
Khouvine, Tomoda, *Compt. rend.*, 1937, **205**, 736, 1414.
Glatthaar, Reichstein, *Helv. Chim. Acta*, 1937, **20**, 1537.

Taiguric Acid.

See Lapachol.

Talatisamine

$C_{22}H_{35}O_4N$ MW, 377

Alkaloid from *Aconitum talassicum*. Cryst. from MeOH. M.p. 144–6°.

B,HCl : 195–6°.

Konovalova, Orekhov, *Bull. soc. chim.*, 1940, **7**, 95.

Talatisidine

$C_{23}H_{37}O_5N$ MW, 407

Alkaloid from *Aconitum talassicum*. Prisms. M.p. 218–19°. $[\alpha]_D$ − 20° in MeOH.

B,HCl : m.p. 187°.

Picrate : m.p. 161–4° decomp.

Konovalova, Orekhov, *Bull. soc. chim.*, 1940, **7**, 95.

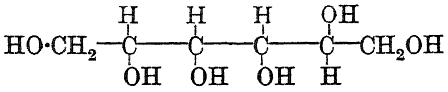
Talatisine

$C_{20}H_{29}O_3N$ MW, 331

Alkaloid from *Aconitum talassicum*. Platelets. M.p. 246·0–246·5°. $[\alpha]_D + 37{\cdot}7°$ in EtOH.
B,HCl: m.p. 256–7°. $[\alpha]_D + 43°$ in MeOH.
B,HI: m.p. 265–6° decomp.
Picrate: m.p. 257–60° decomp.
Triacetyl deriv.: m.p. 211–12°.

Konovalova, Orekhov, *Bull. soc. chim.*, 1940, **7**, 95.

Talitol



$C_6H_{14}O_6$ MW, 182

d-.

Prisms from EtOH. M.p. 86°. $[\alpha]_D^{18} + 3{\cdot}05°$ in H_2O, lævorotatory on addn. of borax. Sol. H_2O, EtOH. Prac. insol. Et_2O. Sweet taste.
Tribenzylidene deriv.: needles. M.p. 210°. $[\alpha]_D - 40°$ in $CHCl_3$. Prac. insol. EtOH. Insol. H_2O.

l-.

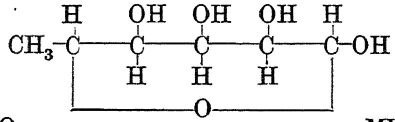
Cryst. from EtOH. M.p. 87–8°. $[\alpha]_D - 2{\cdot}7°$ in H_2O.

dl-.

Needles from AcOEt. M.p. 95–6°. Sol. H_2O. Mod. sol. EtOH.
Tribenzylidene deriv.: cryst. M.p. 205–6°. Spar. sol. EtOH. Prac. insol. H_2O, Et_2O.

Fischer, *Ber.*, 1894, **27**, 1527.
de Bruyn, v. Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151.
Bertrand, Bruneau, *Compt. rend.*, 1908, **146**, 482.
Wolfrom *et al.*, *J. Am. Chem. Soc.*, 1945, **67**, 1226.

Talomethylose



$C_6H_{12}O_5$ MW, 164

d-.

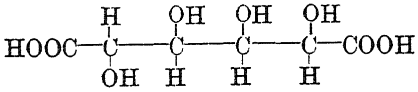
Phenylhydrazine deriv.: m.p. 140–1°.

l-.

Colourless cryst. M.p. 116–18°. $[\alpha]_D^{18} - 20 \pm 2°$ (constant after 3 hours).
p-*Bromophenylhydrazone*: m.p. 145–7°. $[\alpha]_D^{16} - 10 \pm 3°$ after 10 mins.; $+ 4 \pm 3°$ constant.
Methylphenylhydrazone: m.p. 136–7°. $[\alpha]_D^{17} - 13 \pm 3°$ after 45 mins.; $- 12 \pm 3°$ constant.

Gätzi, Reichstein, *Helv. Chim. Acta*, 1938, **21**, 914.
Hamada, Huzita, *Chem. Abstracts*, 1939, **33**, 8572.
Schmutz, *Helv. Chim. Acta*, 1948, **31**, 1719.

Talomucic Acid



$C_6H_{10}O_8$ MW, 210

d-.

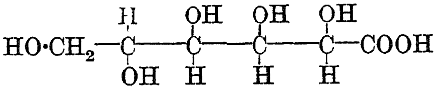
Laminæ from Me_2CO. M.p. 158° decomp. (155–8°). $[\alpha]_D^{20} + 29{\cdot}4°$ in H_2O ($[\alpha]_D + 29° \longrightarrow + 6{\cdot}7°$ in H_2O). Sol. H_2O, hot EtOH. Spar. sol. Me_2CO. Prac. insol. Et_2O, $CHCl_3$, C_6H_6. Partly converted into mucic acid on boiling with Py.Aq.
Diphenylhydrazide: m.p. 185–90°.

l-.

Cryst. from Me_2CO. M.p. 158° decomp. $[\alpha]_D^{20} - 33{\cdot}9°$ in H_2O.
Diphenylhydrazide: m.p. 185°.

Fischer, *Ber.*, 1891, **24**, 3625.
Fischer, Morrell, *Ber.*, 1894, **27**, 391.
Levene, Jacobs, *Ber.*, 1910, **43**, 3145.
Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 198.

Talonic Acid



$C_6H_{12}O_7$ MW, 196

d-.

Cryst. + H_2O from dil. EtOH. M.p. 138–9° (125°). $[\alpha]_D^{25} + 16{\cdot}73° \longrightarrow - 21{\cdot}57°$ in H_2O (18·71° in H_2O). Sol. cold H_2O. Dil. $HNO_3 \longrightarrow$ talomucic acid.
K salt: m.p. 171–2° (169°). $[\alpha]_D^{10} + 2{\cdot}97°$ in H_2O ($[\alpha]_D^{20} + 3°$ in H_2O).
NH_4 salt: m.p. 148°. $[\alpha]_D^{25} + 2{\cdot}9°$ in H_2O.
Brucine salt: $C_{23}H_{26}O_4N_2,C_6H_{12}O_7$. Needles + $3\frac{1}{2}$ (?) H_2O from EtOH. M.p. 95–100°, anhyd. 154–6°. $[\alpha]_D^{20} - 26{\cdot}15°$ in H_2O.
γ-Lactone: m.p. 135–7° (132–4°). $[\alpha]_D^{25} - 34{\cdot}65° \longrightarrow - 28{\cdot}4°$ in H_2O. NaHg $\longrightarrow$ *d*-talose.
Amide: $C_6H_{13}O_6N$. MW, 195. M.p. 121°. $[\alpha]_D^{25} - 13{\cdot}1° \longrightarrow 0°$ in EtOH.
Phenylhydrazide: prisms from EtOH. M.p. 161–2° (159°). $[\alpha]_D^{20} - 24{\cdot}75° \longrightarrow 25{\cdot}1°$ in H_2O ($[\alpha]_D^{25} - 25{\cdot}43°$ in H_2O).
Penta-acetyl: cryst. M.p. 142–4°. $[\alpha]_D + 1{\cdot}8°$ in $CHCl_3$. *Amide*: m.p. 104–6°. $[\alpha]_D + 85{\cdot}4°$ in $CHCl_3$. *Phenylhydrazide*: m.p. 162–3°. $[\alpha]_D + 35°$ in $CHCl_3$.

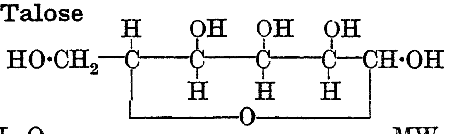
l-.

K salt: m.p. 170–1°. $[\alpha]_D - 1{\cdot}2°$ in H_2O.
Lactone: m.p. 134–6°. $[\alpha]_D + 32{\cdot}4°$.
Amide: cryst. from EtOH. M.p. 119–20°. $[\alpha]_D + 13{\cdot}3°$.

Phenylhydrazide: cryst. from EtOH. M.p. 161–2°. $[\alpha]_D + 26°$.

Hedenburg, Cretcher, *J. Am. Chem. Soc.*, 1927, **49**, 478.
Fischer, *Ber.*, 1891, **24**, 3622.
Fischer, Ruff, *Ber.*, 1900, **33**, 2146.
Cretcher, Renfrew, *J. Am. Chem. Soc.*, 1932, **54**, 1590, 4402.
Brackenbury, Upson, *J. Am. Chem. Soc.*, 1933, **55**, 2512.
Bonnett, Upson, *ibid.*, 1247.
Bosshard, *Helv. Chim. Acta*, 1935, **18**, 485.
Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 203.
Glatthaar, Reichstein, *Helv. Chim. Acta*, 1938, **21**, 3.
Fukunaga, Kubota, *Chem. Abstracts*, 1938, **32**, 4531.

Talose



$C_6H_{12}O_6$ MW, 180

d-.

M.p. 128–30°. $[\alpha]_D^{21}$ about 30° ⟶ 20·6° in H_2O ($[\alpha]_D^{27}$ + 78·7° ⟶ 46·3° in H_2O). Reduces Fehling's. Non-fermentable.

Phenylhydrazone: cryst. M.p. 178° decomp.

p-*Bromophenylhydrazone*: cryst. from EtOH. M.p. 205°.

Phenylosazone: m.p. 202° decomp. (196–7°, 188–91°). Identical with galactosazone.

Methylphenylhydrazone: cryst. from MeOH. M.p. 220–2° (154°).

Benzylphenylhydrazone: yellow laminæ. M.p. 199°.

o-*Nitrophenylhydrazone*: mp. 148·5–149°. $[\alpha]_D^{18}$ + 88·3° in MeOH.

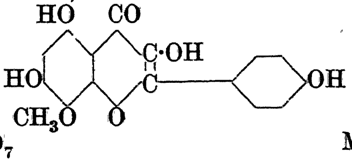
l-.

Syrup. $[\alpha]_D$ — 16·9° in H_2O.

o-*Nitrophenylhydrazone*: cryst. M.p. 144–6°. $[\alpha]_D$ — 77·4°.

v. Braun, Bayer, *Ber.*, 1925, **58**, 2221.
Blanksma, v. Ekenstein, *Chem. Zentr.*, 1908, II, 1584.
Bosshard, *Helv. Chim. Acta*, 1935, **18**, 482.
Komada, *Chem. Abstracts*, 1932, **26**, 4799.
Levene, *J. Biol. Chem.*, 1931, **93**, 631.
Glatthaar, Reichstein, *Helv. Chim. Acta*, 1938, **21**, 3.
Fukunaga, Kubota, *Chem. Abstracts*, 1938, **32**, 4531.

Tambuletin (3 : 5 : 7 : 4′-*Tetrahydroxy*-8-*methoxyflavone*)



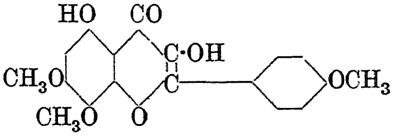
$C_{16}H_{12}O_7$ MW, 316

Found in the seeds of *Zanthoxylum acanthopodium*. Bright yellow needles from AcOH. M.p. 269–70°. Readily sol. hot AcOH. More spar. sol. hot AcOEt, Me_2CO, EtOH. Conc. H_2SO_4 ⟶ bright red changing to brownish-yellow sol. $FeCl_3$ ⟶ dull green col. in EtOH.

Acetyl deriv.: colourless needles from AcOEt. M.p. 140–2°.

Balakrishna, Seshadri, *Chem. Abstracts*, 1948, **42**, 6811, 7296.

Tambulin (3 : 5-*Dihydroxy*-7 : 8 : 4′-*trimethoxyflavone*)



$C_{18}H_{16}O_7$ MW, 344

Found in the fruits of *Zanthoxylum acanthopodium*, D.C. Bright yellow prisms from AcOEt–pet. ether. M.p. 204–5°. $FeCl_3$ ⟶ olive-green col. in EtOH. Aq. alkalis ⟶ brown col.

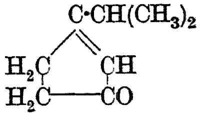
Diacetyl: colourless rectangular rods from AcOEt. M.p. 164–5°.

Balakrishna, Seshadri, *Chem. Abstracts*, 1948, **42**, 7296.

Tanacetone.

See under Thujone.

Tanacetophorone (1-*Isopropylcyclopentenone*-3)



$C_8H_{12}O$ MW, 124

Yellowish-brown oil. B.p. 215–17°, 83·5–84·5°/11 mm. D^{20} 0·9378. n_D^{20} 1·4788. Volatile in steam. Red. ⟶ 3-isopropylcyclopentanone.

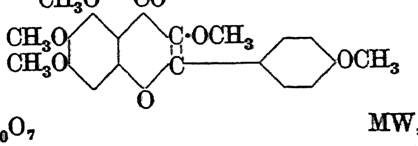
Semicarbazone: plates from EtOH. M.p. 187–8°.

Pringsheim, Bondi, *Ber.*, 1925, **58**, 1415.
Wallach, *Ann.*, 1918, **414**, 221.

Tanacetyl Alcohol.

See Thujyl Alcohol.

Tangeritin (3 : 5 : 6 : 7 : 4′-*Pentamethoxyflavone*)



$C_{20}H_{20}O_7$ MW, 372

Constituent of peel of tangerines. Rods or needles from AcOEt. M.p. 154°. Sol. hot EtOH, hot AcOEt, C_6H_6. Spar. sol. pet. ether. Insol. alkalis. Warm conc. HNO_3 ⟶ blood-red col. Hyd. ⟶ tangeretol + anisic acid.

Boiling HI + phenol ⟶ 3 : 5 : 6 : 7 : 4′-pentahydroxyflavone.

Robinson, Goldsworthy, *J. Chem. Soc.*, 1937, 46.
Nelson, *J. Am. Chem. Soc.*, 1934, **56**, 1392.
Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23***A*, 23.

Tanghiferigenin

$C_{23}H_{32}O_4$ MW, 372

The aglycone from tanghiferin. M.p. 288°.

Frèrejacque, Hasenfratz, *Compt. rend.*, 1946, **223**, 642.

Tanghiferin

$C_{32}H_{46-48}O_9$ MW, 574(576)

Isolated from *Tanghinia venenifera*. Cryst. from EtOH. M.p. 245°. $[\alpha]_D - 64°$ in EtOH. Insol. H_2O. Hyd. ⟶ thevetose.

Acetyl deriv.: cryst. from EtOH. M.p. 231°. $[\alpha]_D - 45{\cdot}5°$ in EtOH.

Frèrejacque, Hasenfratz, *Compt. rend.*, 1946, **223**, 642.
Helfenberger, Reichstein, *Helv. Chim. Acta*, 1952, **35**, 1503.

Tanghinin

$C_{32}H_{46-48}O_{10}$ MW, 590(592)

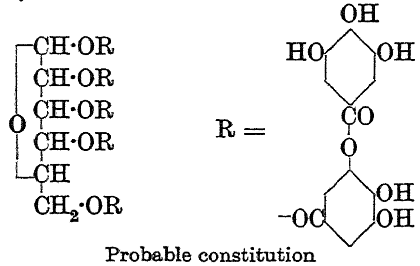
Isolated from *Tanghinia venenifera*. Cryst. as pentahydrate. At 110° the hemihydrate, m.p. 130°, $[\alpha]_D - 79°$ in EtOH, is formed. Hyd. ⟶ thevetose.

Acetyl deriv.: m.p. 188–9°. $[\alpha]_D - 85°$ in EtOH.

Benzoyl deriv.: m.p. 148° (203–5°). $[\alpha]_D - 60°$ ($- 56° \pm 2°$) in EtOH.

Frèrejacque, Hasenfratz, *Compt. rend.*, 1946, **222**, 149.
Helfenberger, Reichstein, *Helv. Chim. Acta*, 1952, **35**, 1503.

Tannic Acid (*Tannin, gallotannin, Chinese tannin, gallotannic acid, penta-*(m-*digalloyl*)-*glucose*)



Probable constitution

$C_{76}H_{52}O_{46}$ MW, 1700

Constituent of galls from many species of oak, particularly *Quercus lusitanica*, also in galls from *Rhus semilata*. Commercial product is insol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. AcOEt. Sol. H_2O, EtOH. Aq. sol. has astringent taste. $FeCl_3$ ⟶ bluish-black col. or ppt. Gives ppts. with most metallic salts, also with many alkaloids, albumen, gelatin, pyridine and quinoline acetate. Optically active with variable dextrorotation. Active principle in several "tannins" used in leather tanning. Has many industrial applications, *e.g.*, as a mordant in the textile industries.

Russell, *Chem. Reviews*, 1935, **17**, 160.
Takino, *Chem. Abstracts*, 1929, **23**, 2707.
Hepworth, *J. Soc. Chem. Ind.*, 1923, **42**, 41T.
Fischer, Bergmann, *Ber.*, 1919, **52**, 829.
Reichel, *Reichsamt Wirtschaftsausbau, Chem. Ber.*, 1942, 764, (*Chem. Abstracts*, 1947, **41**, 4941).

Tanshinone I

$C_{18}H_{12}O_3$ MW, 276

Pigment from the root of *Salvia miltiorrhiza*. M.p. 231°. Conc. H_2SO_4 ⟶ blue col.

Nakao, Fukushima, *J. Pharm. Soc. Japan*, 1934, **54**, 844.
Wessely, Wang, *Ber.*, 1940, **73**, 19.
Wessely, Bauer, *Ber.*, 1942, **75**, 617.
Takiura, *J. Pharm. Soc. Japan*, 1943, **63**, 41.

Tanshinone II

$C_{19}H_{18}O_3$ MW, 294

Pigment from the root of *Salvia miltiorrhiza*. M.p. 216°. Conc. H_2SO_4 ⟶ green col.

Nakao, Fukushima, *J. Pharm. Soc. Japan*, 1934, **54**, 844.
Wessely, Lauterbach, *Ber.*, 1942, **75**, 958.

Tanshinone III

$C_{19}H_{20}O_3$ MW, 296

Pigment from the root of *Salvia miltiorrhiza*. M.p. 182°. Conc. H_2SO_4 ⟶ brown col.

Nakao, Fukushima, *J. Pharm. Soc. Japan*, 1934, **54**, 844.

Taraxanthin

$C_{40}H_{56}O_4$ MW, 600

Isomeric with violaxanthin. Occurs as ester in flowers of dandelion (*Taraxacum officinale*, Wiggers), yellow coltsfoot (*Tussilago farfara*), buttercup (*Ranunculus acer*), etc. Present in mulberry leaves, and in blood glands and eggs of silkworms. Yellow prisms from MeOH. M.p. 184–5°. $[\alpha]_{Cd}^{20} + 200°$ in AcOEt. Absorption bands in CS_2 at 501, 469 and 441 mμ.

Kuhn, Grundmann, *Ber.*, 1934, **67**, 596.
Karrer, Morf, *Helv. Chim. Acta*, 1932, **15**, 863.
Kuhn, Lederer, *Z. physiol. Chem.*, 1931, **200**, 108.
Karrer, Jucker, Rutschmann, Steinlin, *Helv. Chim. Acta*, 1945, **28**, 1153.

Taraxasterol (*α-Lactucerol*)

$C_{30}H_{50}O$ MW, 426

Triterpene alcohol from roots of *Taraxacum officinale* and flowers of *Anthemis nobilis*. Also occurs as isovalerate in latex of *Calotropis procera* and as acetate in resin (lactucarium) of *Lactuca virosa*. Needles from EtOH. M.p. 221–2° (225·5–226°). $[\alpha]_D^{17} + 95{\cdot}9°$ in $CHCl_3$.

Acetyl: plates from AcOEt. M.p. 256–7°. $[\alpha]_D^{18} + 100{\cdot}5°$ in $CHCl_3$.

Bromoacetyl: needles from AcOEt. M.p. 233–4°.

Benzoyl: needles from Me_2CO or C_6H_6–EtOH. M.p. 240–1°. $[\alpha]_D^{12} + 106{\cdot}8°$ in $CHCl_3$.

p-*Nitrobenzoyl*: needles from $CHCl_3$–EtOH. M.p. 277–8°. $[\alpha]_D^{17} + 98{\cdot}3°$ in $CHCl_3$.

Burrows, Simpson, *J. Chem. Soc.*, 1938, 2042.
Power, Browning, *J. Chem. Soc.*, 1912, **101**, 2411.
Bauer, Brunner, *Arch. Pharm.*, 1938, **276**, 605.
Hesse, Eilbracht, Reicheneder, *Ann.*, 1941, **546**, 233.
Simpson, *J. Chem. Soc.*, 1944, 283.
Zimmermann, *Helv. Chim. Acta*, 1945, **28**, 127; 1946, **29**, 1455.
Lardelli, Jeger, *Helv. Chim. Acta*, 1948, **31**, 813.
Lardelli, Krüsi, Jeger, Ruzicka, *Helv. Chim. Acta*, 1948, **31**, 1815.
Dietrich, Jeger, *Helv. Chim. Acta*, 1950, **33**, 715.

ψ-Taraxasterol (*Heterolupeol*)

$C_{30}H_{50}O$ MW, 426

Triterpene alcohol from roots of *Taraxacum officinale*. Also obtained by isomerisation of taraxasterol. Needles from EtOH or $CHCl_3$–MeOH. M.p. 217–19°. $[\alpha]_D + 50°$ in $CHCl_3$.

Acetyl: plates from AcOEt or $CHCl_3$–MeOH. M.p. 240–1°. $[\alpha]_D + 53°$ in $CHCl_3$.

Benzoyl: needles from Me_2CO. M.p. 274–6°. $[\alpha]_D^{21} + 72°$.

Burrows, Simpson, *J. Chem. Soc.*, 1938, 2042.
Lardelli, Krüsi, Jeger, Ruzicka, *Helv. Chim. Acta*, 1948, **31**, 1159, 1815.
Dietrich, Jeger, *Helv. Chim. Acta*, 1950, **33**, 715.

Taraxerol (*Skimmiol*)

$C_{30}H_{50}O$ MW, 426

Triterpene alcohol from rhizomes of *Taraxacum officinale*, and from bark of *Litzea dealbata*, *Alnus incana*, Linn., *Alnus glutinosa*, Linn., *Skimmia japonica*, Thunb., and *Tilia cordata*. Plates from $CHCl_3$–MeOH. M.p. 282–3°. $[\alpha]_D$ 0° in $CHCl_3$.

Acetyl: needles from $CHCl_3$–MeOH. M.p. 304–5°. $[\alpha]_D + 9°$ in $CHCl_3$.

Benzoyl: needles from $CHCl_3$–MeOH. M.p. 292–3°. $[\alpha]_D + 37°$ in $CHCl_3$.

Tribromoacetyl: needles from $CHCl_3$. M.p. 257–8°.

Burrows, Simpson, *J. Chem. Soc.*, 1938, 2042.
Takeda, *J. Pharm. Soc. Japan*, 1941, **61**, 117; 1942, **62**, 390; 1943, **63**, 193, 197.
Koller, Hiestand, Dietrich, Jeger, *Helv. Chim. Acta*, 1950, **33**, 1050.

Taraxol

$C_{30}H_{46}O_3$ MW, 454

Triterpene alcohol from rhizomes of *Taraxacum officinale*. Needles from C_6H_6–EtOH. M.p. above 360°. $[\alpha]_D^{14} + 78{\cdot}6°$ in $CHCl_3$.

Monoacetyl: plates from C_6H_6. M.p. 299–301° decomp. $[\alpha]_D^{14} + 93{\cdot}9°$ in $CHCl_3$.

Oxide: needles from MeOH.Aq. M.p. 261–261·5°. *Acetyl*: plates from C_6H_6. M.p. 294–7°.

Burrows, Simpson, *J. Chem. Soc.*, 1938, 2042.

Tariric Acid (*5-Heptadecyne-1-carboxylic acid*)

$CH_3{\cdot}[CH_2]_{10}{\cdot}C{:}C{\cdot}[CH_2]_4{\cdot}COOH$

$C_{18}H_{32}O_2$ MW, 280

Occurs as glyceride in fruit of *Picramnia-Arten* (Tariri). Cryst. from EtOH. M.p. 50·5°. Red. ⟶ stearic acid. Ox. ⟶ lauric and glutaric acids. Br in cold $CHCl_3$ ⟶ dibromide. I in AcOH ⟶ di-iodide. Gives insol. Ag salt.

Dibromide: cryst. mass. M.p. 32°.

Di-iodide: needles from EtOH. M.p. 48·5°.

Tetrabromide: *see* 5 : 5 : 6 : 6-Tetrabromostearic Acid.

Arnaud, Hasenfratz, *Compt. rend.*, 1911, **152**, 1604.
Arnaud, Posternak, *Compt. rend.*, 1909, **149**, 220.
Lumb, Smith, *J. Chem. Soc.*, 1952, 5032.

Taroxylic Acid (*5 : 6-Diketostearic acid*)

$CH_3{\cdot}[CH_2]_{10}{\cdot}CO{\cdot}CO{\cdot}[CH_2]_4{\cdot}COOH$

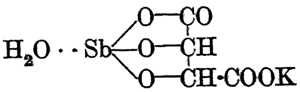
$C_{18}H_{32}O_4$ MW, 312

Pale yellow plates. M.p. 98°. Very sol. boiling EtOH. Spar. sol. cold EtOH. Insol. H_2O. Alkali salts sol. H_2O.

Dioxime: needles. M.p. 166–7°. Very sol. boiling EtOH. Insol. H_2O.

Arnaud, *Compt. rend.*, 1902, **134**, 548.

Tartar emetic (*Potassium antimonyl tartrate*)



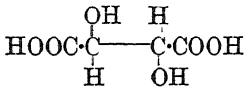
$C_4H_4O_7KSb$ MW, 325

Rhombic cryst. D 2·607. Sol. 3 parts of H_2O at 100°, 25 parts at 15°. One of the few soluble salts of antimony. Used in medicine in small doses: poisonous in larger quantities. Used for mordanting textiles in conjunction with tannic acid.

Reihlen, Hezel, *Ann.*, 1931, **487**, 213.
Chemnitius, *Chem. Abstracts*, 1930, **24**, 2832.
Davies, U.S.P. 2,391,297, (*Chem. Abstracts*, 1946, **40**, 1284).

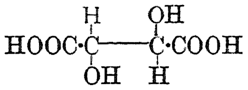
Tartaric Acid (*Dihydroxysuccinic acid*)

Dextro-



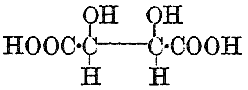
I

Laevo-



II

Meso-



III

Racemic = I + II.

$C_4H_6O_6$ MW, 150

Dextro-.

Ordinary tartaric acid. Occurs in plants, partly free and partly as K, Ca or Mg salts. The KH salt occurs in grape juice. Prisms. M.p. 170° (169°, 168–70°). D_4^7 1·7594, D_4^{18} 1·759, D_4^{20} 1·7598. 100 Parts H_2O dissolve 115·04 parts at 0°, 139·44 parts at 20°, 147·44 parts at 25°, 195·0 parts at 50°, 258·05 parts at 75° and 343·35 parts at 100°. 1 Part dissolves in 2·06 parts 80% EtOH at 15°. 100 Parts 90% EtOH contain 29·146 parts at 15°. 100 Parts abs. EtOH contain 20·385 parts at 15°. 100 Parts Et_2O contain 0·393 parts at 15°. Sol. Me_2CO. $[\alpha]_D^{20}$ + 11·98° (20% sol. in H_2O), $[\alpha]_D^{16}$ + 13·1° (15% sol. in H_2O), $[\alpha]_D$ + 0·47° in MeOH. Heat of comb. C_p 261·75 Cal. k (first) = $1{\cdot}3 \times 10^{-3}$ at 25° ($1{\cdot}02 \times 10^{-3}$, $0{\cdot}97 \times 10^{-3}$); (second) = 69×10^{-6} at 25° (97×10^{-6}, 41×10^{-6}, 45×10^{-6}, $34{\cdot}3 \times 10^{-6}$). Heat $\longrightarrow$ chars with evolution of CO_2 and CO, and odour of burnt sugar (other products include acetaldehyde, acetone, acetic, formic, and pyruvic acids). On boiling with HCl.Aq. or dil. H_2SO_4 or 30% NaOH it is converted into racemic and mesotartaric acids. H_2SO_4 at 100° $\longrightarrow$ CO_2, CO and SO_2. H_2O_2 (+ ferrous salt) $\longrightarrow$ dihydroxymaleic acid (Fenton's reaction). $MnO_2 + H_2SO_4 \longrightarrow$ H·COOH + CO_2. Reacts with cold $KMnO_4$ with difficulty, but rapidly on warming. Reduces $NH_3.AgNO_3$. HI $\longrightarrow$ *d*-malic acid + succinic acid. $HNO_3 + H_2SO_4 \longrightarrow$ di-nitrate of tartaric acid. Prevents the pptn. of CuO and other metallic oxides from alk. sol. Gives yellow col. with $FeCl_3$. Addn. of aq. $CaCl_2$ to neutral sol. pptes. Ca salt, sol. AcOH and cold alkalis.

Mono-NH_4 salt: cryst. Triboluminescent. Sol. 45·6 parts H_2O at 15°. $[\alpha]_D^{15}$ + 25·55° in H_2O.

Di-NH_4 salt: cryst. Triboluminescent. $[\alpha]_D^{15}$ + 34·6° in H_2O. Spar. sol. EtOH.

Mono-Na salt: cryst. + H_2O. Triboluminescent. $[\alpha]_D^{19}$ + 21·8° in H_2O. Sol. H_2O.

Di-Na salt: cryst. + $2H_2O$. Triboluminescent. Sol. 3·46 parts H_2O at 6°, 2·28 parts at 24°, 1·5 parts at 42·5°.

$NaNH_4$ salt: cryst. + $4H_2O$. Triboluminescent. 100 parts aq. sol. at 0° contain 21·2 parts cryst. salt.

Mono-K salt: argol, tartar, cream of tartar (in order of increasing purity). Cryst. Triboluminescent. 100 c.cs. saturated aq. sol. contain 0·370 gm. at 0°, 0·843 gm. at 25°, 1·931 gm. at 50°, 5·850 gm. at 100°.

Di-K salt: cryst. + $\frac{1}{2}H_2O$. Triboluminescent. D_4^{20} 1·984. Sol. 0·75 part H_2O at 2°, 0·66 part at 14°, 0·63 part at 23°, 0·47 part at 64°.

KNH_4 salt: cryst. + $\frac{1}{2}H_2O$. Readily sol. H_2O.

KNa salt: Rochelle Salt. Cryst. + $4H_2O$. Triboluminescent. Sol. 1·70 parts H_2O at 6°.

Potassium antimonyl tartrate: *see* Tartar emetic.

$Ca(C_4H_5O_6)_2$: cryst. + $2H_2O$. 100 parts H_2O dissolve 0·710 part anyhd. salt at 15·6°. More sol. hot H_2O.

$CaC_4H_4O_6$: cryst. + $4H_2O$. Occurs in many plants. 100 gm. H_2O dissolve 0·0185 gm. at 18°, 0·02948 gm. at 25° of hydrated salt. More sol. hot H_2O. Sol. hot alkalis.

$AgC_4H_5O_6$: cryst. + H_2O. Sol. hot aq. tartaric acid.

$Ag_2C_4H_4O_6$: cryst. powder. 100 gm. H_2O dissolve 0·2012 gm. at 18°, 0·2031 gm. at 25°. Insol. EtOH.

Me ester: $C_5H_8O_6$. MW, 164. Prisms + H_2O from H_2O. M.p. 76° (75–7°). 1 part dissolves in 4 parts H_2O at room temp. Sol. AcOEt, Me_2CO. Spar. sol. Et_2O. $[\alpha]_D^{18}$ + 18·71° in H_2O. $k = 4{\cdot}6 \times 10^{-4}$ at 25°.

Di-Me ester: *see* Dimethyl tartrate.

Et ester: $C_6H_{10}O_6$. MW, 178. Prisms. M.p. about 90°. Deliquescent. Sol. H_2O, EtOH. Insol. Et_2O. $[\alpha]_D$ + 21·8° in H_2O.

Di-Et ester: *see* Diethyl tartrate.

Dipropyl ester: $C_{10}H_{18}O_6$. MW, 234. B.p. 303°, 181°/23 mm., 171–2°/17 mm. (173–4°/

17 mm.). D^{20} 1·1344, D^{100} 1·0590, D_4^{20} 1·1390. Sol. H_2O and most org. solvents. $[\alpha]_D^{20}$ + 12·44° in H_2O. *Diacetyl*: m.p. 31°. B.p. 313°, 195–7°/13 mm. $[\alpha]_D^{14}$ + 7·04° in EtOH. *Dibenzoyl*: m.p. 45·5°. B.p. 234°/7 mm. $[\alpha]_{5461}^{17\cdot5}$ − 78·16° in Py.

Di-isopropyl ester: b.p. 275°, 157–8°/16 mm. D^{20} 1·1300, D^{100} 1·0537. $[\alpha]_D^{20}$ + 14·886°. *Diacetyl*: prisms. M.p. 33°. $[\alpha]_D^{20}$ + 5·9° in EtOH.

Dibutyl ester: $C_{12}H_{22}O_6$. MW, 262. Prisms. M.p. 22–22·5°. B.p. 200–3°/18 mm., 178°/12 mm. D^{15} 1·098. $[\alpha]_D^{15}$ + 11·3° in EtOH. *Diacetyl*: b.p. 214°/20 mm. $D^{15\cdot5}$ 1·096. $[\alpha]_D^{22}$ + 8·8° in EtOH. *Dibenzoyl*: m.p. 43°. B.p. 250°/1·3 mm. $[\alpha]_{5461}^{15}$ − 57·62° in Py.

Di-isobutyl ester: m.p. 68°. B.p. 323–5°, 197°/23 mm., 183°/11 mm., 157°/3·5 mm. D^{100} 1·0145. $[\alpha]_D$ + 11·8° in EtOH. *Diacetyl*: b.p. 322–6°, 196–7°/12 mm. $D^{16\cdot5}$ 1·096. $[\alpha]_D^{14}$ + 10·51° in EtOH. *Dibenzoyl*: b.p. about 240°/3 mm. $D^{17\cdot1}$ 1·3360. $[\alpha]_D^{22}$ − 48·86° in EtOH.

*Di-*d*-amyl ester*: $C_{14}H_{26}O_6$. MW, 290. B.p. 208°/20 mm. D_4^{20} 1·0636. $[\alpha]_D^{20}$ + 17·73°.

*Di-*dl*-amyl ester*: b.p. 208°/20 mm. (215–20°/10–25 mm.). D_4^{20} 1·0637. $[\alpha]_D^{20}$ + 14·10°.

Di-isoamyl ester: b.p. 195°/16 mm.

Diallyl ester: $C_{10}H_{14}O_6$. MW, 230. B.p. 191°/20 mm., 171°/10 mm.

Diphenyl ester: $C_{16}H_{14}O_6$. MW, 302. Needles. M.p. 101–2°. Sol. hot EtOH, Et_2O, glycerol. Insol. H_2O.

Dibenzyl ester: $C_{18}H_{18}O_6$. MW, 330. M.p. about 50°. B.p. 250–70°/4 mm. $D^{72\cdot2}$ 1·2036. *Dibenzoyl*: needles from EtOH. M.p. 76–7°. $[\alpha]_D^{18}$ + 6·2° in Me_2CO, + 41·7° in C_6H_6.

Diphenacyl ester: $C_{20}H_{18}O_8$. MW, 386. M.p. 130°.

Anhydride: $C_4H_4O_5$. MW, 132. *Dibenzoyl*: m.p. 173°. $[\alpha]_D^{20}$ + 141·98° in Me_2CO.

Amide: *see* Tartramidic Acid.

Diamide: *see under* Tartramidic Acid.

Dihydrazide: needles. M.p. 182·5–183°. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. $[\alpha]_D^{20}$ + 97·1° in H_2O. *B,2HCl*: cryst. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. N : N-*Diacetyl*: needles from dil. EtOH. M.p. 216°. Sol. H_2O, AcOH. Spar. sol. EtOH. Insol. Et_2O.

Di-azide: cryst. M.p. 66° decomp. Sol. H_2O, EtOH, Me_2CO. Mod. sol. Et_2O. Insol. $CHCl_3$, C_6H_6, ligroin.

Anilide: *see* Tartranilic Acid.

Dianilide: *see under* Tartranilic Acid.

Mono-Me ether: $C_5H_8O_6$. MW, 164. Prisms from Et_2O. M.p. 174°. $[\alpha]_D$ + 45·4° in H_2O.

Di-Me ether: *see* Dimethoxysuccinic Acid.

Di-Et ether: *see* Diethoxysuccinic Acid.

Diacetyl: cryst. + $3H_2O$ from Et_2O. M.p. 58°. Sol. H_2O, EtOH. Spar. sol. $CHCl_3$, C_6H_6. $[\alpha]_D^{22}$ − 23·04° in H_2O. Hyd. by warm aq. alkalis.

Dibenzoyl: cryst. + H_2O from H_2O. M.p. 90° (88–9°), anhyd. 138–40°. $[\alpha]_D^{18}$ − 115·78° in EtOH, $[\alpha]_D^{20}$ − 118·51° in MeOH. Sol. EtOH. Less sol. $CHCl_3$. Spar. sol. cold H_2O, C_6H_6.

Dietrich, *Jahresber. Fortschr. Chem.*, 1878, 1136.
Hölbling, *Chem. Zentr.*, 1896, II, 1068.
Gladyss, D.R.P., 116,090, (*Chem. Zentr.*, 1901, I, 69).
McKenzie, *J. Chem. Soc.*, 1915, **107**, 440.
Voss, *Chem.-Ztg.*, 1921, **45**, 309, 335, 360, 411.
Patterson, *J. Chem. Soc.*, 1913, **103**, 173.
Freundler, *Ann. chim. phys.*, 1894, **3**, 445.
Dyson, *Chemist & Druggist*, 1932, **116**, 668.
Bijvoet, Peerdeman, van Bommel, *Nature*, 1951, **168**, 271.

Laevo-.

Physical properties identical with those of the *dextro*-acid. $[\alpha]_D$ in H_2O of same magnitude but opposite in sign to that of the *dextro*-acid.

Di-Me ester: *see* Dimethyl tartrate.

Di-Et ester: *see* Diethyl tartrate.

Dibenzoyl: cryst. + H_2O. M.p. 85°. $[\alpha]_D^{24}$ + 103·7°.

Di-Me ether: *see* Dimethoxysuccinic Acid.

Marckwald, *Ber.*, 1896, **29**, 42.
Ladenburg, *Ann.*, 1909, **364**, 232.
Parck, *Chem. Abstracts*, 1926, **20**, 1219.
Dale, Rice, *J. Am. Chem. Soc.*, 1933, **55**, 4984.
Kiliani, *Ber.*, 1911, **44**, 112 (*Footnote* 3).
Haskins, Hudson, *J. Am. Chem. Soc.*, 1939, **61**, 1266.
Walton, *J. Soc. Chem. Ind.*, 1945, **64**, 219.

dl-. Racemic Acid.

Cryst. anhyd. from H_2O above 73° or from EtOH. M.p. 206° (205–6°). Cryst. + H_2O, m.p. 203–4°. Loses H_2O at 100°. Less sol. H_2O than dextro- and laevo-acids. 100 Parts H_2O dissolve 9·23 parts hydrated acid at 0°, 14·00 parts at 10°, 20·6 parts at 20°, 24·61 parts at 25°, 29·10 parts at 30°, 59·54 parts at 50°, 184·91 parts at 100°. Sol. 48 parts cold EtOH. Sol. in EtOH at 15° to extent of 2·08%, in Et_2O 1·08%. Heat of comb. C_v (hydrated) 278·4 Cal., (anhyd.) 279·5 Cal. k (first) = 10·2 × 10^{-4} at 25° (10·3 × 10^{-4}, 11 × 10^{-4}, 9·7 × 10^{-4}, 9·6 × 10^{-4}); (second) = 4·0 × 10^{-5} at 25° (2·8 × 10^{-5}, 3·94 × 10^{-5}). Heat with HCl.Aq. at 130–40° or in alk. sol. ⟶ partly to mesotartaric acid.

Mono-NH_4 salt: prisms. Triboluminescent. Sol. 100 parts H_2O at 20°. More sol. boiling H_2O. Insol. EtOH.

Di-NH_4 salt: prisms. Sol. H_2O. Prac. insol. EtOH.

Mono-Na salt: exists hydrated in several modifications. Loses H_2O at 100°. Triboluminescent. Decomp. at 219°. Sol. 11·3 parts H_2O at 19°. More sol. boiling H_2O. Insol. EtOH.

Di-Na salt: prisms. Triboluminescent. Sol. 2·63 parts H_2O at 25°. Insol. EtOH.

$NaNH_4$ salt: prisms + H_2O. Decomposes below 27° into d- and l-$NaNH_4$ tartrates, and above 35° into Na racemate and NH_4 racemate.

Mono-K salt: prisms. Triboluminescent. Sol. 180 parts H_2O at 19°, 139 parts at 25°, 14·3 parts at 100°.

Di-K salt: plates + $2H_2O$. Sol. 0·97 part H_2O at 25°. Prac. insol. EtOH.

KNa salt: prisms + $3H_2O$. M.p. 100°. 100 gms. of saturated aq. sol. contains 36·66 gms. of anhyd. salt at 9·7°, 47·97 gms. at 29·5°. Also prisms or plates + $4H_2O$.

$CaC_4H_4O_6$: cryst. + $4H_2O$. Loses H_2O at 200°. Prac. insol. cold H_2O. More sol. hot H_2O. Insol. AcOH. Sol. HCl.Aq. but pptd. by NH_3.

Me ester: prisms. Sol. H_2O, EtOH. Spar. sol. Et_2O. Hyd. by boiling H_2O.

Di-Me ester: *see* Dimethyl tartrate.

Et ester: prisms. Sol. H_2O, EtOH. Insol. Et_2O.

Di-Et ester: *see* Diethyl tartrate.

Dipropyl ester: m.p. 25°. B.p. 286°/765 mm., 167°/11 mm. D_4^{20} 1·1256.

Di-isopropyl ester: m.p. 34°. B.p. 275°/765 mm., 154°/12 mm. D_4^{20} 1·1166.

Dibutyl ester: b.p. 320°/765 mm., 185°/12 mm. D_4^{18} 1·0879.

Di-isobutyl ester: m.p. 58°. B.p. 311°/768·5 mm., 195°/13 mm. D_4^{20} 1·0160.

Di-d-amyl ester: $C_{14}H_{26}O_6$. MW, 290. B.p. 201–2°/16 mm. (215–25°/10–25 mm.). D_4^{20} 1·064. *n* 1·4501. $[\alpha]_D^{20}$ + 3·37°.

Dibenzoyl: m.p. 112–13°.

Dinitrile: $C_4H_4O_2N_2$. MW, 112. *Diacetyl*: plates or prisms from AcOH. M.p. 97–8°.

Anhydride: *dibenzoyl*, m.p. 182°.

Di-Me ether: *see* Dimethoxysuccinic Acid.

Campbell, Slotin, Johnston, *J. Am. Chem. Soc.*, 1933, **55**, 2604.
Newman, Riley, *J. Chem. Soc.*, 1933, 45.
Hollemann, *Organic Syntheses*, Collective Vol. I, 484.
Pasteur, *Ann. chim. phys.*, 1850, **28**, 79.
Campbell, *J. Chem. Soc.*, 1929, 1111.

Meso-.

Plates + H_2O. M.p. anhyd. 140°. D_4^{20} 1·666. Sol. 0·8 part H_2O at 15°. *k* (first) = $6·0 \times 10^{-4}$ at 25° ($6·3 \times 10^{-4}$); (second) = $1·4 \times 10^{-5}$ at 25°. Heat with H_2O at 175°, with aq. HCl at 130–40° or with aq. alkalis ⟶ partly to racemic acid.

Mono-K salt: needles. Sol. 8 parts H_2O at 19°.

$CaC_4H_4O_6$: prisms + $3H_2O$. Loses H_2O on standing, second H_2O at 100°, third H_2O at 170°. Sol. 600 parts boiling H_2O. Prac. insol. AcOH.

Di-Me ester: *see under* Dimethyl tartrate.

Di-Et ester: *see under* Diethyl tartrate.

Dibutyl ester: m.p. 48–50°.

Di-d-amyl ester: b.p. 203–4°/17 mm. D_4^{20} 1·0658. n_D^{20} 1·4530. $[\alpha]_D^{20}$ + 4·77°.

Dinitrile: plates or prisms from Et_2O. M.p. about 131° decomp. Sol. H_2O, EtOH, Et_2O. Spar. sol. $CHCl_3$. Prac. insol. CS_2. Not very stable. *Diacetyl*: prisms from Et_2O or AcOH. M.p. 75–7°.

Anhydride: *dibenzoyl*, m.p. 207–8°.

Di-Me ether: *see* Dimethoxysuccinic Acid.

Winther, *Z. physik. Chem.*, 1906, **56**, 507.
Pollak, *Monatsh.*, 1894, **15**, 469.
Milas, U.S.P. 2,414,385, (*Chem. Abstracts*, 1947, **41**, 5895).
Noller, *Science*, 1945, **102**, 508.

Tartaric Dialdehyde.

See Tartraldehyde.

Tartraldehyde (*Tartaric dialdehyde, dihydroxysuccindialdehyde*)

```
      H    OH
      |    |
OHC·C——C·CHO
      |    |
      OH   H
```

$C_4H_6O_4$ MW, 118

l-.

Monophenylhydrazone: $[\alpha]_D^{18}$ − 183°.

α-*Di-phenylhydrazone*: platelets from EtOH. Aq. Decomp. at 177–9°. $[\alpha]_D^{20}$ − 100° in Py.

β-*Di-phenylhydrazone*: platelets from AcOH–Py. Decomp. at 195°. $[\alpha]_D$ − 2° in Py.

Bergmann, *Ber.*, 1921, **54**, 2651.

Tartramide.

See under Tartramidic Acid.

Tartramidic Acid (*Tartaric monoamide*)

```
HO·CH·CO·NH2
   |
HO·CH·COOH
```

$C_4H_7O_5N$ MW, 149

d-.

Cryst. from H_2O. M.p. 171–2°. $[\alpha]_D^{13}$ + 63·7° in H_2O.

$Ca(C_4H_6O_5N)_2$: cryst. + $6H_2O$. Sol. H_2O. Insol. EtOH. $[\alpha]_D^{18}$ + 59·5° in H_2O.

Et ester: $C_6H_{11}O_5N$. MW, 177. Plates. M.p. 136–7°.

Amide: $C_4H_8O_4N_2$. MW, 148. *d*-Tartramide, diamide of tartaric acid. Cryst. M.p. 195° decomp. Spar. sol. MeOH, EtOH. Insol. Et_2O, C_6H_6. $[\alpha]_D^{20}$ + 106·5° in H_2O, + 144° in MeOH. *Di-Me ether*: *see under* Dimethoxysuccinic Acid. *Dibenzoyl*: needles from EtOH. M.p. 240°.

Di-Me ether: *see under* Dimethoxysuccinic Acid.

Anilide: *see under* Tartranilic Acid.

Weerman, *Rec. trav. chim.*, 1917, **37**, 45.
Grote, *Ann.*, 1864, **130**, 203.
Frankland, Slator, *J. Chem. Soc.*, 1903, **83**, 1354.

Tartranilic Acid (*Tartaric monoanilide*)

HO·CH·CO·NH·C_6H_5
HO·CH·COOH

$C_{10}H_{11}O_5N$ MW, 225

d-.

Plates. M.p. 180° decomp. Mod. sol. H_2O, EtOH. (Needles from AcOH. M.p. 194°.) $[\alpha]_D^{15}$ + 114·7° in MeOH.

NH_4 salt: needles. $[\alpha]_D^{15}$ + 102·2° in H_2O.

Na salt: needles. M.p. 226°. Spar. sol. EtOH. $[\alpha]_D^{15}$ + 101·3° in H_2O.

Aniline salt: cryst. M.p. 149–50°.

Me ester: $C_{11}H_{13}O_5N$. MW, 239. Needles from AcOH. M.p. 175°. Sol. EtOH. Spar. sol. H_2O. $[\alpha]_D^{15}$ + 106° in MeOH.

Et ester: $C_{12}H_{15}O_5N$. MW, 253. Plates from H_2O. M.p. 151–2°. (Needles, m.p. 163°.) Sol. hot H_2O. $[\alpha]_D^{15}$ + 102·4° in MeOH.

Propyl ester: $C_{13}H_{17}O_5N$. MW, 267. Plates from EtOH. M.p. 161°. $[\alpha]_D^{15}$ + 99·1° in MeOH.

Isobutyl ester: $C_{14}H_{19}O_5N$. MW, 281. Needles from EtOH. M.p. 158°. $[\alpha]_D^{15}$ + 92·6° in MeOH.

Isoamyl ester: $C_{15}H_{21}O_5N$. MW, 295. Needles from EtOH. M.p. 139°. $[\alpha]_D^{15}$ + 89·2° in MeOH.

Amide: $C_{10}H_{12}O_4N_2$. MW, 224. Anilide of tartramidic acid. Plates. M.p. 226°. $[\alpha]_D^{15}$ + 139° in H_2O, + 153° in MeOH.

Anilide: $C_{16}H_{16}O_4N_2$. MW, 300. Dianilide of tartaric acid. Prisms from MeOH or needles from EtOH. M.p. 275° (250° decomp., 255–6° decomp., 263–4° decomp.). Sol. Py. Spar. sol. Et_2O, EtOH. Prac. insol. C_6H_6, AcOH. Insol. H_2O. $[\alpha]_D^{15}$ + 259° in Py ($[\alpha]_D^{20}$ + 246·5° in Py), + 206° in MeOH. *Acetyl*: needles from EtOH. M.p. 148°. *Diacetyl*: needles from dil. EtOH. M.p. 214–15°. Sol. EtOH, Et_2O, $CHCl_3$, Me_2CO, C_6H_6. Prac. insol. ligroin. Insol. H_2O. *Tetra-acetyl*: needles + 2EtOH from EtOH. M.p. 137°.

Di-Me ether: *see under* Dimethoxysuccinic Acid.

Di-Et ether: *see under* Diethoxysuccinic Acid.

Tingle, Bates, *J. Am. Chem. Soc.*, 1909, **31**, 1240.
Polikier, *Ber.*, 1891, **24**, 2959.
Bischoff, Nastvogel, *Ber.*, 1890, **23**, 2047.
Arppe, *Ann.*, 1855, **93**, 355.
Casale, *Gazz. chim. ital.*, 1917, **47**, 277.

Tartronic Acid (*Hydroxymalonic acid*)

$$HO\cdot CH\langle^{COOH}_{COOH}$$

$C_3H_4O_5$ MW, 120

Prisms + H_2O from H_2O. Loses H_2O at 60°. M.p. varies with rate of heating; 156–8° (10° per minute), 141–2° (1° per minute). Sol. H_2O, EtOH. Spar. sol. Et_2O. Anhyd. acid sol. Et_2O. Sublimes. *k* (first) = 5 × 10^{-3} at 25°. Forms cryst. salts.

Di-Me ester: $C_5H_8O_5$. MW, 148. M.p. 44·5–45°. Sol. $CHCl_3$, Et_2O. Spar. sol. pet. ether.

Di-Et ester: $C_7H_{12}O_5$. MW, 176. F.p. − 2·5°. B.p. 222–5° (218–19°), 120·5–121°/15 mm. D^{15} 1·152. *Et ether*: b.p. 228°. *Acetyl*: b.p. 235–45°, 158–63°/60 mm., 138°/17–18 mm. $D_{15}^{19·5}$ 1·131.

Monoamide: $C_3H_5O_4N$. MW, 119. Needles or prisms from H_2O. M.p. about 160° decomp. Sol. EtOH. Spar. sol. cold H_2O. Prac. insol. Et_2O.

Diamide: $C_3H_6O_3N_2$. MW, 118. Needles from EtOH.Aq. M.p. 195–6° (198°). Mod. sol. hot H_2O. Spar. sol. cold H_2O, EtOH.

Et ether: $C_5H_8O_5$. MW, 148. Prisms from pet. ether. M.p. 123–5°. Decomp. at 135°. Sol. H_2O, EtOH. Spar. sol. C_6H_6, ligroin.

Wislicenus, Münzesheimer, *Ber.*, 1898, **31**, 552.
Conrad, Brückner, *Ber.*, 1891, **24**, 2997.
Behrend, Prüsse, *Ann.*, 1918, **416**, 233.
Filippo, *Rec. trav. chim.*, 1910, **29**, 115.
Bak, *Ann.*, 1939, **537**, 286.

Tartronylurea.

See Dialuric Acid.

Taurine (*Aminoethylsulphonic acid, amino-ethanesulphonic acid, ethylaminesulphonic acid*)

$H_2N\cdot CH_2\cdot CH_2\cdot SO_3H$

$C_2H_7O_3NS$ MW, 125

Occurs mainly combined. Free in lungs and flesh extract of oxen. In shark blood; liver, spleen and kidney of ray; in muscle, oysters, molluscs, etc. Columns from H_2O. M.p. 328° (317° decomp.). Sol. 15·5 parts H_2O at 12°, 1·7 parts H_2O at 100°. Insol. EtOH. Neutral in dil. sol. Acid in conc. sol. Heat of comb. C_v 382·2 Cal., C_p 382·9 Cal. Stable to boiling conc. acids. Not esterified readily. Phenol + hypochlorites ⟶ blue col. MeOH + KOH + MeI ⟶ taurobetaine. Hg salt used for identification and separation.

N-*Me*: $C_3H_9O_3NS$. MW, 139. Prisms. M.p. 241–2°. Very sol. H_2O. Insol. EtOH, Et_2O.

N-*Di-Me*: $C_4H_{11}O_3NS$. MW, 153. Prisms from MeOH. M.p. 315–16°. Very sol. H_2O, AcOH. Insol. EtOH, Et_2O.

N-*Et*: $C_4H_{11}O_3NS$. MW, 153. Prisms from H_2O. M.p. 147°.

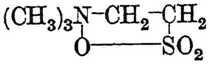
N-*Di-Et*: $C_6H_{15}O_3NS$. MW, 181. Plates from EtOH. M.p. 151°.

N-*Allyl*: $C_5H_{11}O_3NS$. MW, 165. Prisms from EtOH. M.p. 190–5°.

Amide: *hydrochloride*, plates from EtOH. M.p. 133°.

Cortese, *J. Am. Chem. Soc.*, 1936, **58**, 191.
Teroaka, *Z. physiol. Chem.*, 1925, **145**, 238.
James, *J. prakt. Chem.*, 1885, **31**, 414.
Rumpf, *Bull. soc. chim.*, 1938, **5**, 871.
Cortese, *Organic Syntheses*, 1938, XVIII, 77.
Carr, Shutt, *Trans. Faraday Soc.*, 1939, **35**, 584.
Desseigne, *Bull. soc. chim.*, 1942, **9**, 786.
Goldberg, *J. Chem. Soc.*, 1943, 4.
Heath, Piggott, *J. Chem. Soc.*, 1947, 1481.
Schick, Degering, *Ind. Eng. Chem.*, 1947, **39**, 906.

Taurobetaine (*Trimethyltaurine*)



$C_5H_{13}O_3NS$ MW, 167

Prisms from H_2O. Does not melt below 250°. Very sol. H_2O. Insol. EtOH, Et_2O. Neutral reaction. Sweet taste. $Ba(OH)_2 \longrightarrow$ trimethylamine + isethionic acid.

Brieger, *Z. physiol. Chem.*, 1882, **7**, 36.

Taurocarbamic Acid (2-*Ureidoethane*-1-*sulphonic acid, sulphoethylurea*)

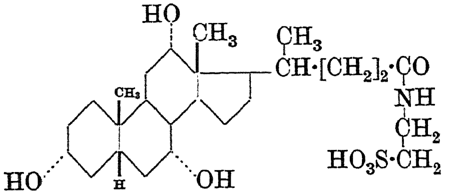
$H_2N{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}SO_3H$

$C_3H_8O_4N_2S$ MW, 168

Prisms from EtOH.Aq. M.p. 182° decomp. Very sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. $Ba(OH)_2$ or HCl on heating $\longrightarrow$ taurine. Forms cryst. Ba and Ag salts.

Lippich, *Z. physiol. Chem.*, 1910, **68**, 292.

Taurocholic Acid



$C_{26}H_{45}O_7NS$ MW, 515

Constituent of bile. Amorph. powder. M.p. about 125° decomp. Very sol. H_2O, hot EtOH. Spar. sol. Et_2O, AcOEt. Very hygroscopic. $[\alpha]_D^{20}$ + 38·8° in EtOH.Aq.

Na salt: needles or plates + $1\frac{1}{2}H_2O$ from H_2O. M.p. anhyd. 180°. Boiling $H_2O \longrightarrow$ isomeric form, cryst. + $2H_2O$, m.p. 235°. Both forms have identical solubilities and rotations.

Ba salt: plates or needles + $5H_2O$ from H_2O. M.p. 225–7° decomp. Very sol. H_2O, hot EtOH. Spar. sol. Et_2O, AcOEt. $[\alpha]_D^{20}$ + 25·6°.

Kazuno, Yamasaki, *Z. physiol. Chem.*, 1934, **224**, 160.
Tanaka, *Z. physiol. Chem.*, 1933, **220**, 39.
Hammarsten, *Z. physiol. Chem.*, 1904, **43**, 127.

Tazettine (*Ungernine*)

$C_{18}H_{21}O_5N$ MW, 331

Constituent of bulbs of *Narcissus tazetta*. After high vac. sublimation, m.p. 210–11°. Sol. EtOH. Spar. sol. Et_2O. $[\alpha]_D^{16}$ + 150·4 in $CHCl_3$. $H_2SO_4 \longrightarrow$ brownish-red col. Zn dust dist. $\longrightarrow$ phenanthridine. $KMnO_4 \longrightarrow$ hydrastic acid.

Acetyl deriv.: cryst. from Et_2O-pet. ether. M.p. 125–126·5°.

Methiodide: m.p. 220° decomp.

Picrate: yellow cryst. from EtOH. M.p. 205–8° decomp.

Perchlorate: m.p. 105–8° decomp. $[\alpha]_D^{15}$ + 109·6° in MeOH.

Robinson, *Ann. Rev. Biochem.*, 1935, **4**, 507.
Späth, Kahovec, *Ber.*, 1934, **67**, 1501.
Norkina, Orechoff, *Ber.*, 1936, **69**, 500.
Späth, Orechoff, Kuffner, *ibid.*, 2446.
Clemo, Felton, *Chemistry and Industry*, 1952, 807.

Tecomin.

See Lapachol.

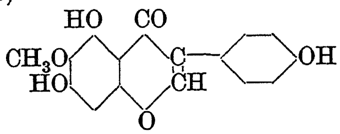
Tectochrysin.

See under Chrysin.

Tectoquinone.

See under 2-Methylanthraquinone.

Tectorigenin (5 : 7 : 4′-*Trihydroxy*-6-*methoxyisoflavone*)



$C_{16}H_{12}O_6$ MW, 300

Occurs as glycoside, tectoridin, in the rhizomes of *Balameanda chinensis* and *Iris tectorum*, Maxim. Yellow plates from EtOH. M.p. 227° decomp.

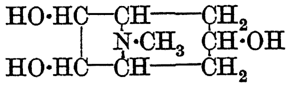
Triacetyl: prisms. M.p. 187°.

Tribenzoyl: plates. M.p. 238°.

Di-Me ether: $C_{18}H_{16}O_6$. MW, 328. M.p. 188°. *Acetyl*: m.p. 213–14°.

Asahina, Shibata, Ogawa, *J. Pharm. Soc. Japan*, 1928, **48**, 1087.
Shibata, *Chem. Zentr.*, 1927, II, 839.
Shriner, Stephenson, *J. Am. Chem. Soc.*, 1942, **64**, 2737.

Teloidine (3 : 6 : 7-*Trihydroxytropane*, *tropantriol*-3 : 6 : 7)



$C_8H_{15}O_3N$ MW, 173

Needles + $1H_2O$ from Me_2CO.Aq. M.p. 169–70° (168–9°). Very sol. H_2O, EtOH. Spar. sol. most cold org. solvents.

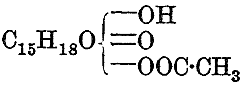
B,HCl: prisms from EtOH. Does not melt below 300°.

B,HBr: plates and needles from EtOH. M.p. 295° decomp.

B,HAuCl₄: yellow plates + $\frac{1}{2}H_2O$ from H_2O. M.p. 225° decomp.

King, *J. Chem. Soc.*, 1919, **115**, 487.
Pyman, Reynolds, *J. Chem. Soc.*, 1908, **93**, 2079.
Schöpf, Arnold, *Ann.*, 1947, **558**, 109.

Tenulin

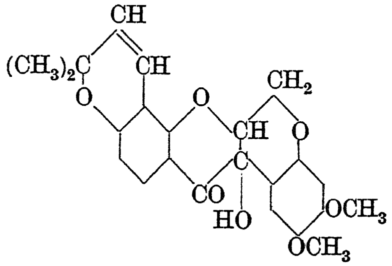


$C_{17}H_{22}O_5$ MW, 306

Constituent of *Helenium tenuifolium*, *H. elegans*, etc. Cryst. from H_2O. M.p. 194–6°. $[\alpha]_D^{20}$ − 21·7° in EtOH. Fish poison. Mild sternutatory action. H(+ Pt) ⟶ dihydrotenulin, m.p. 182°. Br ⟶ dibromotenulin, m.p. 124–5°. Alkalis ⟶ isotenulin, m.p. 162°.

Clark, *J. Am. Chem. Soc.*, 1940, **62**, 597; 1939, **61**, 1836.
Ungnade, Hendley, *J. Am. Chem. Soc.*, 1948, **70**, 3921.

Tephrosin (*Hydroxydeguelin, allotephrosin, isoallotephrosin*)



$C_{23}H_{22}O_7$ MW, 410

Constituent of derris root, cubé root (*Lonchocarpus nicou*) and leaves of *Cracca vogeli*. Prisms from MeOH–$CHCl_3$. M.p. 198°. Ox. ⟶ tephrosin-dicarboxylic acid.

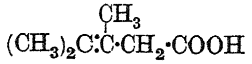
Acetyl: cryst. from MeOH.Aq. M.p. 200°.

Clark, *J. Am. Chem. Soc.*, 1933, **55**, 759; 1932, **54**, 3000; 1931, **53**, 729.
Takei, Mayujima, Ono, *Chem. Abstracts*, 1933, **27**, 2954.
Clark, Claborn, *J. Am. Chem. Soc.*, 1932, **54**, 4454.
Butenandt, Hilgetag, *Ann.*, 1932, **495**, 172.
Boam, Cahn, Stuart, *J. Soc. Chem. Ind.*, 1937, **56**, 91.

Teraconic Acid.

See Isopropylidenesuccinic Acid.

Teracrylic Acid (2 : 3-*Dimethyl-2-pentene-5-carboxylic acid, 2-isopropylidene-butyric acid*)



$C_7H_{12}O_2$ MW, 128

Liq. B.p. 218°. Spar. sol. H_2O.

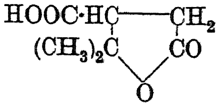
Et ester: $C_9H_{16}O_2$. MW, 156. Liq. with fruity odour. B.p. 189–91°.

Fittig, Kraft, *Ann.*, 1881, **208**, 79.

Tereanilic Acid.

See 2 : 5-Diaminoterephthalic Acid.

Terebic Acid (2 : 2-*Dimethylparaconic acid*, 3 : 3-*dimethylbutyrolactone-2-carboxylic acid*)



$C_7H_{10}O_4$ MW, 158

d-.

$[\alpha]_D^{25}$ + 13·2° in Me_2CO.

l-.

M.p. 201–5° decomp. $[\alpha]_D^{25}$ − 13·2° in Me_2CO. Sol. 9 gm. per litre H_2O at 25°.

dl-.

Cryst. from EtOH. M.p. 176° (175°). Sol. boiling H_2O, warm EtOH. Sol. 13·6 gm. per litre H_2O at 25°. Heat of comb. C_v 778·4 Cal. $k = 2{\cdot}65 \times 10^{-4}$ at 25°.

Me ester: $C_8H_{12}O_4$. MW, 172. B.p. 148–9°/17 mm.

Et ester: $C_9H_{14}O_4$. MW, 186. B.p. 273–5°, 145–7°/15 mm.

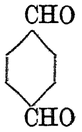
Chloride: $C_7H_9O_3Cl$. MW, 176·5. B.p. 143°/12 mm.

Anilide: cryst. from EtOH. M.p. 176°.

p-*Toluidide*: cryst. from EtOH or C_6H_6. M.p. 186–7°.

Barbier, Locquin, *Bull. soc. chim.*, 1913, **13**, 231.
Simonsen, *J. Chem. Soc.*, 1907, **91**, 186.
Rupe, Zweidler, *Helv. Chim. Acta*, 1940, **23**, 1035.
Fredga, *Chem. Abstracts*, 1948, **42**, 123.
Fichter, Schetty, *Helv. Chim. Acta*, 1937, **20**, 1304.

Terephthalaldehyde (*Terephthaldialdehyde*, 1 : 4-*dialdehydobenzene*)



$C_8H_6O_2$ MW, 134

Needles from H_2O. M.p. 116°. B.p. 245–8°/771 mm. Sol. 5000 parts cold H_2O, 80 parts hot H_2O. Sol. Et_2O. Very sol. EtOH. Sol. alkalis, pptd. by acids. Sublimes. Difficultly volatile in steam. $K_2Cr_2O_7 + H_2SO_4$ ⟶ *p*-aldehydo-

benzoic acid. $Ac_2O + H_2SO_4 \longrightarrow$ tetra-acetate.

Dioxime: terephthalaldoxime. $C_8H_8O_2N_2$. MW, 164. Cryst. M.p. 200°. Spar. sol. H_2O. Very sol. EtOH, Et_2O. *Di-Et ether*: cryst. M.p. 55°. *Diacetyl*: cryst. M.p. 155°.

Dihydrazone: yellowish-white cryst. from EtOH. M.p. 165° decomp.

Monophenylhydrazone: orange-yellow needles from Et_2O. M.p. 152–4°.

Di-phenylhydrazone: yellow scales from AcOH or acetoacetic ester. M.p. 278° decomp.

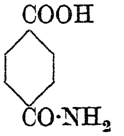
Di-p-*nitrophenylhydrazone*: cryst. from $PhNO_2$. Sinters at 272°, m.p. 281°.

Adams, Bullock, Wilson, *J. Am. Chem. Soc.*, 1923, **45**, 521.
Rosenmund, Zetzsche, *Ber.*, 1921, **54**, 2888.
Thiele, Günther, *Ann.*, 1906, **347**, 110.
Snell, Weissberger, *Organic Syntheses*, 1940, XX, 92.

Terephthalaldehydic Acid.

See p-Aldehydobenzoic Acid.

Terephthalamic Acid (*Terephthalic acid monoamide*)



$C_8H_7O_3N$ MW, 165

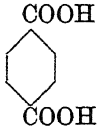
Does not melt below 300°. Begins to sublime at 250°. Insol. hot H_2O and most org. solvents. Hyd. $\longrightarrow$ terephthalic acid.

Me ester: $C_9H_9O_3N$. MW, 179. Cryst. from hot H_2O. M.p. 201°.

Nitrile: *see under p*-Cyanobenzoic Acid.

Kattwinkel, Wolffenstein, *Ber.*, 1904, **37**, 3222.

Terephthalic Acid (p-*Phthalic acid, benzene-1 : 4-dicarboxylic acid*)



$C_8H_6O_4$ MW, 166

Needles. Sublimes without melting at about 300°. Sol. 67,000 parts cold H_2O. Insol. Et_2O, EtOH, AcOH, $CHCl_3$. Sol. hot EtOH. Heat of comb. C_p 770·9 Cal., C_v 771·2 Cal. Gives spar. sol. Ca, Sr and Ba salts. Red. $\longrightarrow$ *cis-* and *trans*-hexahydroterephthalic acids.

Mono-Me ester: $C_9H_8O_4$. MW, 180. Needles from hot H_2O. M.p. about 230°. Sublimes.

Di-Me ester: $C_{10}H_{10}O_4$. MW, 194. Needles from Et_2O. M.p. 141–2°. Sol. 300 parts hot H_2O. Spar. sol. cold EtOH. Sublimes. Volatile in steam.

Mono-Et ester: $C_{10}H_{10}O_4$. MW, 194. Plates from C_6H_6. M.p. 171°.

Di-Et ester: $C_{12}H_{14}O_4$. MW, 222. Prisms from EtOH or pet. ether. M.p. 44°. B.p. 302°, 142°/2 mm. D_{45}^{45} 1·1098. Sol. Et_2O. Very sol. cold EtOH. Insol. H_2O.

Mono-propyl ester: $C_{11}H_{12}O_4$. MW, 208. M.p. 127–9°.

Dipropyl ester: $C_{14}H_{18}O_4$. MW, 250. Needles or plates from C_6H_6. M.p. 31°. B.p. 158°/4 mm. Very sol. hot EtOH, Et_2O.

Mono-isopropyl ester: needles from hot C_6H_6. M.p. 166°. Very sol. EtOH, Et_2O. Insol. H_2O.

Di-isopropyl ester: plates. M.p. 55–6°.

Mono-butyl ester: $C_{12}H_{14}O_4$. MW, 222. M.p. 122–4°.

Dibutyl ester: $C_{16}H_{22}O_4$. MW, 278. Needles. M.p. 16°. B.p. 180°/4 mm.

Mono-isobutyl ester: m.p. 151–4°.

Di-isobutyl ester: plates. M.p. 55°. B.p. 180°/6 mm.

Di-tert.-*butyl ester*: prisms from MeOH. M.p. 118°.

Di-l-*menthyl ester*: $C_{28}H_{42}O_4$. MW, 442. Needles from EtOH. M.p. 77–8°. $[\alpha]_D^{20}$ − 102·6° in $CHCl_3$.

Diphenyl ester: $C_{20}H_{14}O_4$. MW, 318. Needles from EtOH. M.p. 191°.

Monochloride: $C_8H_5O_3Cl$. MW, 184·5. Needles from C_6H_6. Does not melt below 300°.

Dichloride: $C_8H_4O_2Cl_2$. MW, 203. Needles or plates from ligroin. M.p. 83–4°.

Dibromide: $C_8H_4O_2Br_2$. MW, 292. Needles from pet. ether. M.p. 85°.

Mono-amide: *see* Terephthalamic Acid.

Diamide: $C_8H_8O_2N_2$. MW, 164. Plates from AcOH. Needles from H_2O. Does not melt below 250°.

Di-l-*naphthylamide*: needles from $PhNO_2$. Sinters at 325°, m.p. 334–5°.

Di-diphenylamide: needles from xylene. Sinters at 268°, m.p. 272–3° decomp.

Di-azide: plates from Me_2CO. M.p. 110°.

Di-hydrazide: needles from H_2O. Does not melt below 300°.

Mono-nitrile: *see p*-Cyanobenzoic Acid.

Di-nitrile: 1 : 4-dicyanobenzene. $C_8H_4N_2$. MW, 128. Needles from H_2O or MeOH. M.p. 222°. Very sol. hot AcOH. Spar. sol. EtOH, Et_2O. Insol. cold H_2O. Sublimes.

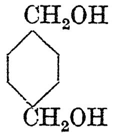
Dianilide: needles from acetoacetic ester or $PhNO_2$. M.p. 334–7°.

Carré, Libermann, *Compt. rend.*, 1934, **199**, 1422.
Smith, *J. Am. Chem. Soc.*, 1921, **43**, 1920.
Rosenmund, Zetzsche, *Ber.*, 1921, **54**, 2888.
Lyons, Reid, *J. Am. Chem. Soc.*, 1917, **39**, 1740.
Cohen, Pennington, *J. Chem. Soc.*, 1918, **113**, 63.
Koelsch, *Organic Syntheses*, 1946, XXVI, 95.

Terephthalophenone.

See *p*-Dibenzoylbenzene.

Terephthalyl Alcohol (p-*Xylylene glycol*, ω-*dihydroxy*-p-*xylene*)



$C_8H_{10}O_2$ MW, 138

Needles. M.p. 115–16°. Sol. H_2O, EtOH, Et_2O.

Mono-Me ether: $C_9H_{12}O_2$. MW, 152. B.p. 152°/16 mm. D_4^{17} 1·076. n_D^{17} 1·529. *Acetyl*: b.p. 150°/16 mm. D_4^{20} 1·080. n_D^{20} 1·505. *Phenylurethane*: m.p. 62°.

Di-Me ether: $C_{10}H_{14}O_2$. MW, 166. Liq. B.p. 235°, 124°/18 mm. D_4^{16} 1·013. n_D^{16} 1·503.

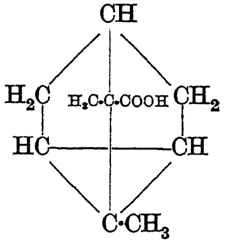
Mono-Et ether: $C_{10}H_{14}O_2$. MW, 166. Liq. B.p. 250–2°, 154°/16 mm. D_4^{17} 1·047. n_D^{17} 1·520. Sol. EtOH, Et_2O. Insol. H_2O.

Di-Et ether: $C_{12}H_{18}O_2$. MW, 194. Liq. B.p. 251–2°/734 mm., 134°/15 mm. D_4^{18} 0·976. n_D^{18} 1·493.

Dibenzyl ether: $C_{22}H_{22}O_2$. MW, 318. M.p. 67°.

Quelet, *Bull. soc. chim.*, 1933, **53**, 222.

Teresantalic Acid



$C_{10}H_{14}O_2$ MW, 166

Occurs in sandalwood oil. Prisms from EtOH. M.p. 158°. B.p. 157–8°/20 mm. Sol. 1270 parts H_2O at 20°. $[\alpha]_D^{20}$ − 76·6° in C_6H_6. $k = 0{\cdot}69 \times 10^{-5}$ at 22·8°. Volatile in steam. Stable to $KMnO_4$. Forms a hydrochloride. Dil. H_2SO_4 ⟶ santene.

Ca salt: cryst. + $1\frac{1}{2}H_2O$ from H_2O. Decomp. at 100°.

Hydrochloride: cryst. from MeOH. M.p. 199°. Spar. sol. pet. ether.

Me ester: $C_{11}H_{16}O_2$. MW, 180. Oil. B.p. 88·5–89·5°/13 mm. D_4^{20} 1·0305. $[\alpha]_D^{20}$ − 60·79°. Na + EtOH ⟶ teresantalol. *Hydrochloride*: two forms. (i) Leaflets from EtOH.Aq. M.p. 68°. B.p. 125–7°/10 mm. $[\alpha]_D^{20}$ + 9·22° in C_6H_6. (ii) Oil. *Hydrobromide*: oil. F.p. −10°. M.p. 20°.

Chloride: $C_{10}H_{13}OCl$. MW, 184·5. B.p. 100–4°/14 mm.

Anilide: b.p. 204–5°/11 mm.

Lactone: m.p. 196°.

Asahina, Ishidate, Momose, *Ber.*, 1935, **68**, 83.

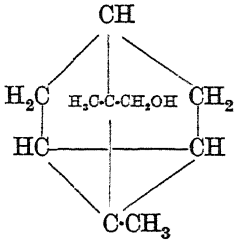
Ruzicka, Liebl, *Helv. Chim. Acta*, 1926, **9**, 140.

Rupe, Tomi, *Ber.*, 1916, **49**, 2556.

Steiger, Rupe, *Helv. Chim. Acta*, 1937, **20**, 1117.

Guha, Bhattacharyya, *J. Indian Chem. Soc.*, 1944, **21**, 377.

Teresantalol



$C_{10}H_{16}O$ MW, 152

d-.

Constituent of Indian sandalwood oil. Prisms from pet. ether–C_6H_6. M.p. 115°. B.p. 95–8°/9 mm. $[\alpha]_D$ + 12·1° in EtOH. Sublimes.

Acetyl: b.p. 102–3°/9–10 mm. D^{20} 1·019. n_D^{20} 1·470. $[\alpha]_D$ + 21°.

dl-.

Prisms from C_6H_6. M.p. 118°. B.p. 97–8°/10 mm. Stable to cold $KMnO_4$.

Asahina, Ishidate, *Ber.*, 1935, **68**, 952.

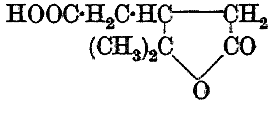
Semmler, Bartelt, *Ber.*, 1907, **40**, 3103.

Guha, Bhattacharyya, *J. Indian Chem. Soc.*, 1944, **21**, 271.

Terpane.

See *p*-Menthane.

Terpenylic Acid (3 : 3-*Dimethylbutyrolactone-2-acetic acid*)



$C_8H_{12}O_4$ MW, 172

Plates or prisms + $1H_2O$ from H_2O. M.p. 57°, anhyd. 90°. Very sol. hot H_2O. Mod. sol. cold H_2O. Sublimes at 130–40°. Ox. ⟶ terebic acid. HI (+ P) ⟶ 2-isopropylglutaric acid.

Me ester: $C_9H_{14}O_4$. MW, 186. B.p. 145–7°/15 mm.

Et ester: $C_{10}H_{16}O_4$. MW, 200. M.p. 37·5°. B.p. 305°, 174–7°/15 mm.

Lawrence, *J. Chem. Soc.*, 1899, **75**, 530.

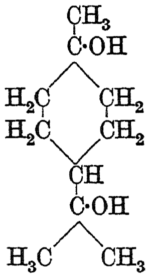
Wallach, *Ann.*, 1893, **277**, 118.

Fittig, Levy, *Ann.*, 1890, **256**, 109.

Terphenyl.

See Diphenylbenzene.

Terpin (p-*Menthandiol*-1 : 8, *terpinol*)



$C_{10}H_{20}O_2$ MW, 172

Cis :

Cryst. M.p. 105·5°. B.p. 258°. Absorbs moisture from the air. Heat of comb. C_v 1454·4 Cal. Dist. over Al_2O_3 at 200–80° ⟶ dipentene. H (+ Ni) ⟶ *p*-cymene. Oxalic acid ⟶ *d*-terpineol.

Diformyl : viscous oil. B.p. 176–7°/40 mm. D^{27} 1·067.

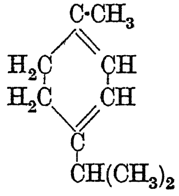
Diacetyl : b.p. 145°/14 mm.

Trans :

Prisms or plates from AcOEt. M.p. 156–8°. B.p. 263–5°. Very sol. EtOH. Spar. sol. H_2O, Et_2O, AcOEt.

Aschan, *Chem. Zentr.*, 1919, I, 284.
Wallach, *Ann.*, 1906, **350**, 154.
Machado, *Chem. Abstracts*, 1943, **36**, 1441.
Trimble, *Ind. Eng. Chem.*, 1941, **33**, 660.
Ipatieff, Pines, *J. Am. Chem. Soc.*, 1944, **66**, 1120; 1945, **67**, 1226.

α-Terpinene ($\Delta^{1,3}$-p-*Menthadiene*)



$C_{10}H_{16}$ MW, 136

Constituent of many essential oils. Oil with lemon odour. B.p. 173·5–174·8°/755 mm. D_4^{19} 0·8375. $n_D^{19·7}$ 1·477. Resinifies on keeping. Heat of comb. C_v 1469·9 Cal.

Di-hydrochloride : terpinene dihydrochloride. Cryst. from MeOH. M.p. 53–4°. B.p. 108–9°/10 mm.

Di-hydrobromide : terpinene dihydrobromide. Plates from MeOH or AcOH. M.p. 58–9°.

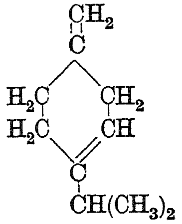
Di-hydriodide : terpinene dihydriodide. Cryst. from MeOH. M.p. 76°.

Nitrosite : prisms. M.p. 155°. *Benzoate* : m.p. 77–8°.

Maleic Anhydride add. comp. : cryst. from pet. ether. M.p. 62° (66–7°).

Richter, Wolff, *Ber.*, 1930, **63**, 1714.
Wallach, *Ann.*, 1889, **252**, 133; 1887, **241**, 316; 1887, **239**, 35.
Briggs, Sutherland, *J. Org. Chem.*, 1942, **7**, 397.

β-Terpinene ($\Delta^{3,1(7)}$-p-*Menthadiene*)



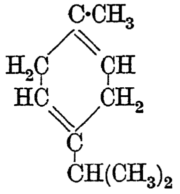
$C_{10}H_{16}$ MW, 136

Does not occur naturally. Oil. B.p. 173–4°. D^{22} 0·838. n_D^{22} 1·4754. Readily oxidised in air. HCl ⟶ terpinene dihydrochloride.

Tetrabromide : prisms from AcOEt. M.p. 154–5°.

Wallach, *Ann.*, 1908, **362**, 288.

γ-Terpinene ($\Delta^{1,4}$-p-*Menthadiene*)



$C_{10}H_{16}$ MW, 136

Constituent of a few essential oils. B.p. 183°, 72·5°/18 mm. $[\alpha]_D^{25}$ + 36·0°. D_4^{20} 0·849. $n_D^{14·5}$ 1·4765. Heat of comb. C_v 1468·9 Cal., C_p 1472 Cal. HCl ⟶ terpinene dihydrochloride.

Tetrabromide : plates from pet. ether. M.p. 129–30° (125·5–126·5°).

Nitrosochloride : m.p. 111°.

Nitrolpiperidide : plates. M.p. 149°.

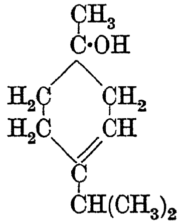
Nitrosate : needles from AcOH–MeOH. M.p. 116° decomp.

Richter, Wolff, *Ber.*, 1930, **63**, 1714.
Briggs, Sutherland, *J. Org. Chem.*, 1948, **13**, 6.

Terpinene-terpin.

See p-Menthandiol-1 : 4.

(1)-Terpinenol (Δ^3-p-*Menthenol*-1, 4-*methyl*-1-*isopropylcyclohexenol*-4)

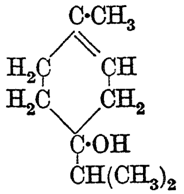


$C_{10}H_{18}O$ MW, 154

B.p. 208–10°. D^{18} 0·9265. n_D^{18} 1·4781. $KMnO_4$ ⟶ *p*-menthantriol-1 : 3 : 4.

Wallach, Meister, *Ann.*, 1908, **362**, 269.
Hückel, Kümmel, *J. prakt. Chem.*, 1942, **160**, 74.

(4)-Terpinenol (Δ^1-p-*Menthenol*-4, 1-*methyl*-4-*isopropylcyclohexenol*-4)



$C_{10}H_{18}O$ MW, 154

d-.

Occurs in many essential oils. B.p. 208–10°, 93–6°/1 mm. D^{20} 0·926. n_D^{19} 1·4785. $[\alpha]_D^{11}$ + 24·5°. Dil. $H_2SO_4 \longrightarrow$ *p*-menthandiol-1 : 4. $KMnO_4 \longrightarrow$ *d*-menthantriol-1 : 2 : 4.

Formyl: b.p. 102–6°. D^{20} 0·975. n_D 1·4745.

Nitrosochloride: m.p. 111–12°.

Nitrolpiperidide: m.p. 172–4°.

Phenylurethane: m.p. 71–2°.

1-*Naphthylurethane*: m.p. 107–8° (105·5–106·5°).

dl-.

Constituent of oil from leaf of *Liquidambar formosana*, Hance. B.p. 212–14°, 90°/11 mm. n_D 1·4803.

Nitrolpiperidide: Two forms. (i) Cryst. from EtOH. M.p. 155–6°. (ii) Cryst. from EtOH. M.p. 181–2°. More soluble.

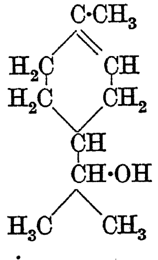
Kafuku, Nozoe, Hata, *J. Chem. Soc. Japan*, 1934, **55**, 224.

Wallach, Meister, *Ann.*, 1908, **362**, 269.

Wallach, *Ber.*, 1907, **40**, 596.

Briggs, Sutherland, *J. Org. Chem.*, 1942, **7**, 397.

α-Terpineol (Δ^1-p-*Menthenol*-8)



$C_{10}H_{18}O$ MW, 154

All forms occur in many essential oils.

d-.

M.p. 36·9°. B.p. 104°/15 mm. $D_4^{21.5}$ 0·9475. n_D 1·4819. $[\alpha]_D^{20}$ + 100·5°.

Nitrosochloride: leaflets from MeOH. M.p. 107–8°.

Nitrolpiperidide: m.p. 151–2°.

Formyl: b.p. 133–6°/40 mm. D_0 0·9989. $[\alpha]_D$ + 16·5°.

Acetyl: b.p. 140°/40 mm. D_0^0 0·9828. $[\alpha]_D$ + 52·5°.

p-*Nitrobenzoyl*: m.p. 139°. $[\alpha]_D^{19.5}$ + 43·9°.

Hydrogen phthaloyl: $[\alpha]_D$ + 36·7°. *Morphine salt*: rhombic cryst. from MeOH. M.p. 142°.

Phenylurethane: needles from MeOH. M.p. 109·5°.

l-.

M.p. 37·0°. B.p. 104°/15 mm. D_4^{25} 0·9364. n_D^{25} 1·48054. $[\alpha]_D^{20}$ − 100·5°.

Nitrosochloride: needles from MeOH.Aq. M.p. 107–8°.

Nitrolpiperidide: m.p. 151–2°.

Formyl: b.p. 135–8°/40 mm. D_0^0 0·9986. $[\alpha]_D$ − 69·4°.

Hydrogen phthaloyl: $[\alpha]_D$ − 36·7°. *Strychnine salt*: needles from EtOH. M.p. 207°.

Phenylurethane: m.p. 109·5°.

dl-.

M.p. 40–41° (35°). B.p. 218·8–219·4°/752 mm., 99–100°/12 mm. D_{20}^{20} 0·935. n_D^{20} 1·4819. Non-misc. with H_2O. Misc. with most org. solvents. Heat of comb. C_v 1475·1 Cal. $KMnO_4 \longrightarrow$ *p*-menthantriol-1 : 2 : 8 and terpenylic acid. H_2O at 250° $\longrightarrow$ dipentene. H·COOH $\longrightarrow$ terpinolene. H(+ Ni) $\longrightarrow$ *p*-cymene, H(+ Pd) $\longrightarrow$ *p*-menthanol-8. $MgCl_2 \longrightarrow$ α-terpinene and dipentene.

Nitrosochloride: needles from AcOEt. Woolly needles from MeOH. M.p. 120–2°.

Nitrolanilide: m.p. 145–6°.

Nitrolpiperidide: m.p. 159–60°.

Me ether: $C_{11}H_{20}O$. MW, 168. B.p. 212°.

Acetyl: b.p. 104–6°/11 mm. D_4^{20} 0·9659. $n_D^{19.7}$ 1·4657.

p-*Nitrobenzoyl*: needles from pet. ether. M.p. 97°.

Hydrogen phthaloyl: rods from AcOH. M.p. 117–18°. *Brucine salt*: rhombic cryst. from MeOH. M.p. 150°.

Phenylurethane: m.p. 113°.

Allophanate: m.p. 121–122·5°.

Ransac, *Chem. Zentr.*, 1932, I, 63.

Fuller, Kenyon, *J. Chem. Soc.*, 1924, **125**, 2304.

Perkin, *J. Chem. Soc.*, 1904, **85**, 665.

Hückel, Nerdel, *Ann.*, 1937, **528**, 57.

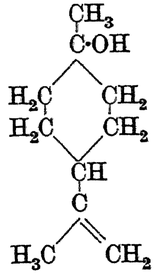
Earl, Hazelwood, *J. Chem. Soc.*, 1937, 374.

Ipatieff, Pines, *J. Am. Chem. Soc.*, 1944, **66**, 1120.

Hückel, Wolowski, *Chem. Ber.*, 1947, **80**, 39.

Naves, Grampoloff, *Helv. Chim. Acta*, 1942, **25**, 1500.

β-Terpineol ($\Delta^{8(9)}$-p-*Menthenol*-1)



$C_{10}H_{18}O$ MW, 154

Does not occur naturally. Oil with odour of hyacinths. Solidifies on cooling to needles, m.p. 32–3°. B.p. 209–10°/752 mm., 90°/10 mm. D_{20}^{20} 0·919. n_D^{20} 1·4747. $KMnO_4$ ⟶ *p*-menthantriol-1 : 8 : 9.

Dibromide: needles from EtOH–Et_2O. M.p. 114–15°.

Nitrosite: needles from Me_2CO.Aq. M.p. 78°.

Nitrosate: needles from EtOH.Aq. M.p. 125°.

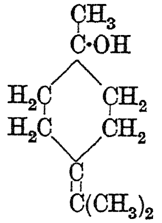
Nitrosochloride: cryst. from EtOH.Aq. M.p. 103°.

Nitrolanilide: m.p. 145–6°.

Phenylurethane: m.p. 85°.

Ransac, *Chem. Zent.*, 1932, I, 63.
Stephen, Helle, *Ber.*, 1902, **35**, 2149.

γ-Terpineol ($\Delta^{4(8)}$-p-*Menthenol*-1)



$C_{10}H_{18}O$ MW, 154

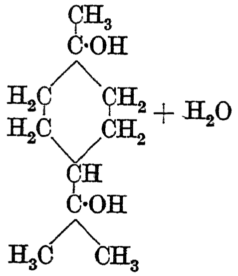
Thick prisms from Et_2O. M.p. 68–70°. Distils without decomp. $KMnO_4$ ⟶ *p*-menthantriol-1 : 4 : 8. H·COOH ⟶ terpinolene.

Dibromide: m.p. 114–15°.

Acetyl: b.p. 110–20°/16 mm. *Dibromide*: m.p. 103°. *Nitrosochloride*: blue leaflets from EtOH. M.p. 82°. *Nitrosobromide*: blue needles from EtOH. M.p. 81–2°.

Baeyer, *Ber.*, 1894, **27**, 443.
Frank, McPherson, *J. Am. Chem. Soc.*, 1949, **71**, 1387.

Terpin Hydrate



$C_{10}H_{20}O_2,H_2O$ MW, 190

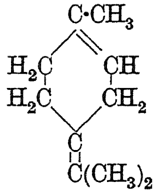
Rhombic cryst. M.p. 123° decomp. ⟶ *cis*-terpin. Eutectic containing 10% hydrate has m.p. 95°. Sol. 250 parts H_2O at 15°, 32 parts at 100°. Sol. 10 parts EtOH at 15°, 2 parts at 78°. Sol. 100 parts Et_2O, 200 parts $CHCl_3$ at 15°. Sol. 1 part of AcOH at b.p. Insol. pet. ether. H_2SO_4 ⟶ orange col. Heat of comb. C_v 1449·1 Cal.

Claus, *Chem. Abstracts*, 1932, **26**, 127.
Aschan, *Chem. Zentr.*, 1919, I, 284.
Dodge, *Chem. Abstracts*, 1940, **34**, 7532.

Terpinol.

See Terpin.

Terpinolene ($\Delta^{1,4(8)}$-p-*Menthadiene*)



$C_{10}H_{16}$ MW, 136

Naturally occurring terpene hydrocarbon. B.p. 186°, 121°/111 mm., 76°/10 mm. Polymerises when dist. at atm. press. Very readily isomerises to terpinene. D_{15}^{15} 0·8633. n_D^{20} 1·4883. HCl ⟶ dipentene dihydrochloride. Ac_2O + H_2SO_4 ⟶ transient pink col.

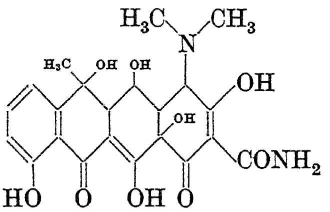
Dibromide: prisms. M.p. 69–70°.

Tetrabromide: two forms. They show marked m.p. depression but give same terpinolene on debromination. (i) Rectangular plates from Me_2CO. M.p. 119°. (ii) Cryst. aggregates from EtOH, Me_2CO or AcOEt. M.p. 122°.

Maleic Anhydride add. comp.: m.p. 182°.

Henry, Paget, *J. Chem. Soc.*, 1931, **134**, 28.
Wallach, *Ann.*, 1909, **368**, 11.
Hultzsch, *Ber.*, 1939, **72**, 1182.
Briggs, Sutherland, *J. Org. Chem.*, 1942, **7**, 397.

Terramycin



$C_{22}H_{24}O_9N_2$ MW, 460

A broad spectrum antibiotic elaborated by *Streptomyces rimosus*. Sinters at 182°. M.p. 184·5–185·5° decomp.

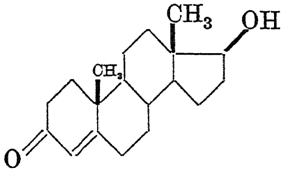
Dihydrate: m.p. 181–2° decomp. $[\alpha]_D^{25}$ − 196·6° in 0·1*N*-HCl (equilibrium value); − 2·1° in 0·1*N*-NaOH (equilibrium value).

B,HCl: needles from MeOH. Platelets from H_2O at 50°.

Na salt: lemon-yellow cryst. from MeOH.

Finlay *et al.*, *Science*, 1950, **111**, 85.
Pasternack *et al.*, *J. Am. Chem. Soc.*, 1951, **73**, 2400.
Regna *et al.*, *J. Am. Chem. Soc.*, 1951, **73**, 4211.
Hochstein, Pasternack, *J. Am. Chem. Soc.*, 1951, **73**, 5008.
Hochstein *et al.*, *J. Am. Chem. Soc.*, 1952, **74**, 3708.

Testane-3α : 17β-diol.
See Aetiocholane-3α : 17β-diol.
Testane-3α-ol-17-one.
See Aetiocholane-3α-ol-17-one.
Testane-3β-ol-17-one.
See Aetiocholane-3β-ol-17-one.
Testosterone (Δ^4-*Androstene*-17β-*ol*-3-*one*)



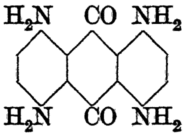
$C_{19}H_{28}O_2$ MW, 288

Hormone obtained from testes extract. Needles from Me_2CO. M.p. 154–154·5°. $[\alpha]_D$ + 109° in EtOH. Produces male characteristics in castrated animals.

Oxime : cryst. from EtOH.Aq. M.p. 222–3°.
Formyl : cryst. from hexane. M.p. 127–9°.
Acetyl : needles from Me_2CO.Aq. M.p. 140–1°. $[\alpha]_D^{20}$ + 87·8° in EtOH.
Propionyl : m.p. 121–3°.
Butyryl : needles from 70% MeOH. M.p. 111–13°.
Isobutyryl : needles from Me_2CO.Aq. M.p. 134–6°.
Palmityl : cryst. from MeOH. M.p. 72–4°.
Stearyl : cryst. from MeOH. M.p. 79–80°.
Benzoyl : m.p. 190°. $[\alpha]_D^{20}$ + 160°.

David, Dingemance, Freud, Laquer, *Z. physiol. Chem.*, 1935, **233**, 281.
Butenandt, Hanisch, *Ber.*, 1935, **68**, 1859.
Ruzicka, Wettstein, *Helv. Chim. Acta*, 1935, **18**, 1264; 1936, **19**, 1141.
Serini, Koster, Strassberger, U.S.P. 2,379,832, (*Chem. Abstracts*, 1945, **39**, 5054).

***epi*-Testosterone.**
See Δ^4-Androstene-17α-ol-3-one.
Δ^5-Testosterone.
See Δ^5-Androstene-17β-ol-3-one.
Tetanthrene.
See Tetrahydrophenanthrene.
Tethracene.
See Tetrahydroanthracene.
Tethracenequinone.
See 1 : 2 : 3 : 4-Tetrahydroanthraquinone.
1 : 4 : 5 : 8-Tetra-aminoanthraquinone



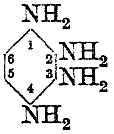
$C_{14}H_{12}O_2N_4$ MW, 268

Reddish-brown needles from EtOH. M.p. 332°. Sol. EtOH and $CHCl_3$ with greenish-blue col., in AcOEt with reddish-blue col., in Me_2CO with blue col. Spar. sol. H_2O with blue col. Cl_2(+ H_2SO_4) ⟶ 1 : 4 : 5 : 8-tetrachloroanthraquinone.

1 : 4 : 5 : 8-N-*Tetra-Me* : $C_{18}H_{20}O_2N_4$. MW, 324. Cryst. Sol. $CHCl_3$, AcOEt with blue col. Sol. HCl with red col. turning violet on dilution.
1 : 4 : 5 : 8-N-*Tetra-acetyl* : cryst. from $PhNO_2$. M.p. 315–20°.

Noelting, Wortmann, *Ber.*, 1906, **39**, 644.
Höchst, D.R.P., 156,803, (*Chem. Zentr.*, 1905, I, 313).
Hall, Hey, *J. Chem. Soc.*, 1948, 736.

1 : 2 : 3 : 4-Tetra-aminobenzene



$C_6H_{10}N_4$ MW, 138

Free base oxidises very rapidly in air.

Nietzki, Geese, *Ber.*, 1899, **32**, 505.

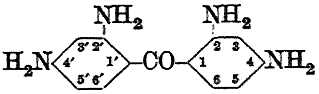
1 : 2 : 3 : 5-Tetra-aminobenzene.
B,3HCl : needles + H_2O.
B,2H₂SO₄ : plates.
N-*Tetra-acetyl* : needles from AcOH. M.p. 245°.

Nietzki, Hagenbach, *Ber.*, 1897, **30**, 539.
Borsche, *Ber.*, 1923, **56**, 1939.

1 : 2 : 4 : 5-Tetra-aminobenzene.
Free base oxidises very readily in air.
B,4HCl : prisms. Sol. H_2O. Spar. sol. HCl.
B,H_2SO_4 : needles. Mod. sol. H_2O.
B_2,3H_2SO_4 : plates. Spar. sol. H_2O.
2 : 4-N-*Diphenyl* : $C_{18}H_{18}N_4$. MW, 290. Needles from C_6H_6. M.p. 207°.
1 : 2 : 4 : 5-N-*Tetraphenyl* : $C_{30}H_{26}N_4$. MW, 442. Needles. M.p. 173–4°.
1 : 2 : 4 : 5-N-*Tetra-acetyl* : needles from AcOH. M.p. 285°.

Nietzki, Hagenbach, *Ber.*, 1887, **20**, 334.
Nietzki, Müller, *Ber.*, 1889, **22**, 440.
Hewitt, Stevenson, *Ber.*, 1898, **31**, 1791.

2 : 4 : 2′ : 4′-Tetra-aminobenzophenone



$C_{13}H_{14}ON_4$ MW, 242

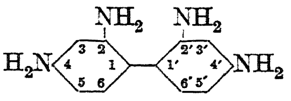
Yellow prisms from EtOH. M.p. 202°. Evolves NH_3 above m.p. Sol. MeOH, Me_2CO, AcOEt. Spar. sol. $CHCl_3$, C_6H_6.

Gulland, Robinson, *J. Chem. Soc.*, 1925, 1499.

3 : 4 : 3′ : 4′-Tetra-aminobenzophenone.
Yellow needles from H_2O. M.p. 217°.

Montagne, *Ber.*, 1915, **48**, 1034.

2 : 4 : 2′ : 4′-Tetra-aminodiphenyl (2 : 2′-*Diaminobenzidine*)



$C_{12}H_{14}N_4$ MW, 214

Leaflets from EtOH.Aq. M.p. 166°. 20% HCl or H_2SO_4 at 200° ⟶ 2 : 7-diaminocarbazole.

2 : 2′-N-*Tetra-Me* : $C_{16}H_{22}N_4$. MW, 270. Needles from EtOH. M.p. 165·5–166°. Sol. C_6H_6. Mod. sol. EtOH. Spar. sol. H_2O, ligroin.

4 : 4′-N-*Tetra-Me* : plates from C_6H_6–ligroin. M.p. 166°. Sol. EtOH, C_6H_6. Spar. sol. Et_2O, ligroin. Insol. H_2O.

2 : 4 : 2′: 4′-N-*Tetra-acetyl* : needles from H_2O. M.p. 285°.

Täuber, *Ber.*, 1890, **23**, 797.
Elbs, Wohlfahrt, *J. prakt. Chem.*, 1902, **66**, 561.
Noelting, Fourneaux, *Ber.*, 1897, **30**, 2940.
Ullmann, Dieterle, *Ber.*, 1904, **37**, 33.

2 : 5 : 2′ : 5′-Tetra-aminodiphenyl.

Needles from toluene. M.p. 168°. Sol. H_2O, EtOH, $CHCl_3$. Spar. sol. Et_2O, C_6H_6. Insol. ligroin. HCl at 190° ⟶ 3 : 6-diaminocarbazole.

Täuber, *Ber.*, 1892, **25**, 130.

3 : 4 : 3′ : 4′-Tetra-aminodiphenyl (3 : 3′-*Diaminobenzidine*).

Plates. Rapidly blackens in air.

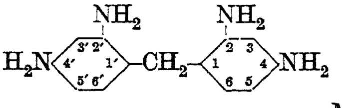
B,4HCl : needles + $2H_2O$. Sol. H_2O.

B,H₂SO₄ : needles. Sol. hot H_2O. Spar. sol. EtOH, Et_2O.

4 : 4′-N-*Tetra-Me* : plates from EtOH. M.p. 168°. Sol. EtOH. Insol. H_2O. $FeCl_3$ + HCl ⟶ violet col.

Brunner, Witt, *Ber.*, 1887, **20**, 1025.
Michler, Pattinson, *Ber.*, 1881, **14**, 2164; 1884, **17**, 118.

2 : 4 : 2′ : 4′-Tetra-aminodiphenylmethane



$C_{13}H_{16}N_4$ MW, 228

Cryst. from H_2O. M.p. 161°. Sol. EtOH. Mod. sol. H_2O. Spar. sol. C_6H_6. Insol. Et_2O.

4-N-*Di-Me* : $C_{15}H_{20}N_4$. MW, 256. Cryst. from toluene. M.p. 188–90°. Sol. EtOH, $CHCl_3$. Insol. H_2O.

4-N-*Me*-4′-N-*Di-Me* : $C_{16}H_{22}N_4$. MW, 270. Cryst. from toluene. M.p. 95°. Sol. EtOH, $CHCl_3$. Insol. H_2O.

4 : 4′-N-*Tetra-Me* : $C_{17}H_{24}N_4$. MW, 284. Needles from EtOH. M.p. 142°. Sol. $CHCl_3$, C_6H_6, hot EtOH. Mod. sol. Et_2O. Insol. H_2O.

4 : 4′-N-*Diacetyl* : needles from EtOH. M.p. 244°.

2 : 4 : 2′ : 4′-N-*Tetra-acetyl* : cryst. from H_2O. Mod. sol. EtOH. Spar. sol. H_2O.

2 : 4 : 2′ : 4′-N-*Tetrabenzoyl* : cryst. from EtOH. M.p. 275°.

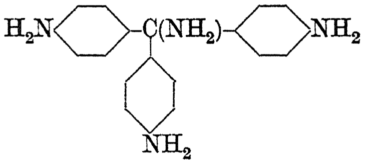
Staedel, *Ann.*, 1883, **218**, 341.
Duval, *Compt. rend.*, 1906, **142**, 342.
Pinnow, *Ber.*, 1894, **27**, 3163.
Bayer, D.R.P., 133,709, (*Chem. Zentr.*, 1902, II, 615).

3 : 4 : 3′ : 4′-Tetra-aminodiphenylmethane.

Plates from H_2O or C_6H_6. M.p. 137–8°. Sol. hot H_2O. Spar. sol. EtOH, C_6H_6. Insol. Et_2O, Me_2CO. $FeCl_3$ + HCl ⟶ dark red col.

Meyer, Rohmer, *Ber.*, 1900, **33**, 257.

α : 4 : 4′ : 4″-Tetra-aminotriphenylmethane



$C_{19}H_{20}N_4$ MW, 304

Yellow prisms. Sol. Py. Spar. sol. common org. solvents.

4 : 4′ : 4″-N-*Hexa-Me* : $C_{25}H_{32}N_4$. MW, 388. Leaflets from C_6H_6–ligroin. M.p. 190·5° decomp. Spar. sol. Et_2O, ligroin.

4 : 4′ : 4″-N-*Hexa-Et* : $C_{31}H_{44}N_4$. MW, 472. Needles from ligroin. M.p. 141·5–142·5°. Sol. Et_2O, C_6H_6. Mod. sol. ligroin.

Villiger, Kopetschni, *Ber.*, 1912, **45**, 2920.
Noelting, Saas, *Ber.*, 1913, **46**, 953.

2 : 3 : 4 : 6-Tetrabromoacetanilide.

See under 2 : 3 : 4 : 6-Tetrabromoaniline.

1 : 1 : 1 : 3-Tetrabromoacetone

$BrCH_2·CO·CBr_3$

$C_3H_2OBr_4$ MW, 374

Needles. M.p. 37–8°. B.p. 258° decomp., 139°/14 mm.

Tetrahydrate : prisms. M.p. 62°.

Mulder, *J. prakt. Chem.*, 1864, **91**, 475.
de Jong, *Rec. trav. chim.*, 1903, **22**, 286.
Dippy, Watson, Yates, *J. Chem. Soc.*, 1931, 2508.

1 : 1 : 3 : 3-Tetrabromoacetone

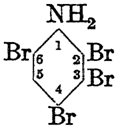
$Br_2CH·CO·CHBr_2$

$C_3H_2OBr_4$ MW, 374

Yellow oil. Decomp. on warming. Alkalis ⟶ bromoform.

Lederer, D.R.P., 98,009.

2 : 3 : 4 : 6-Tetrabromoaniline



$C_6H_3NBr_4$ MW, 409

Cryst. from EtOH. M.p. 118°.

N-*Acetyl* : 2 : 3 : 4 : 6-tetrabromoacetanilide. Needles from C_6H_6. M.p. 228–9°.

N-*Diacetyl* : prisms from pet. ether. M.p. 164°.

$C_6H_3NBr_4,C_6H_3(NO_2)_3$-1 : 3 : 5 : yellow needles. M.p. 107·5–108°.

N-*Thionyl* : yellow needles from C_6H_6. M.p. 78°.

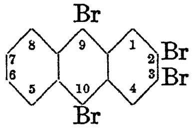
Dains, Kenyon, *J. Am. Chem. Soc.*, 1931, **53**, 2363.
Sudborough, Beard, *J. Chem. Soc.*, 1910, **97**, 782.
Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 50.
Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 57.

2 : 3 : 5 : 6-Tetrabromoaniline.
Needles from EtOH. M.p. 130°.

Claus, *J. prakt. Chem.*, 1895, **51**, 412.

Tetrabromoanisole.
See under Tetrabromophenol.

2 : 3 : 9 : 10-Tetrabromoanthracene



$C_{14}H_6Br_4$ MW, 494

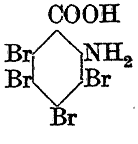
Yellow needles from C_6H_6. M.p. 274° (265–6°). Spar. sol. EtOH, Et_2O, C_6H_6. Sublimes. Ox. ⟶ 2 : 3-dibromoanthraquinone.

Barnett, Cook, *J. Chem. Soc.*, 1935, 1489.
Meyer, Zahn, *Ann.*, 1913, **396**, 174.

2 : 6 : 9 : 10-Tetrabromoanthracene.
Cryst. from CCl_4 or toluene. M.p. 298–300°. Mod. sol. hot toluene. Spar. sol. EtOH, C_6H_6. Ox. ⟶ 2 : 6-dibromoanthraquinone.

Grandmougin, *Compt. rend.*, 1921, **173**, 1176.
Kaufler, Imhoff, *Ber.*, 1904, **37**, 4706.

Tetrabromoanthranilic Acid (3 : 4 : 5 : 6-*Tetrabromo-2-aminobenzoic acid*)

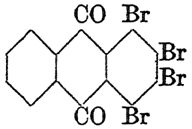


$C_7H_3O_2NBr_4$ MW, 453

Needles from AcOH. M.p. 204–5°. Sol. EtOH, AcOEt. Mod. sol. $CHCl_3$, AcOH, xylene.

Lesser, Weiss, *Ber.*, 1913, **46**, 3942.
Grandmougin, *Ber.*, 1914, **47**, 384.

1 : 2 : 3 : 4-Tetrabromoanthraquinone



$C_{14}H_4O_2Br_4$ MW, 524

Orange-red needles from ligroin. M.p. 200–2°.

Hofmann, *Monatsh.*, 1915, **36**, 820.

The following tetrabromoanthraquinones of unknown constitution have also been described :

(i) Yellow needles from $PhNO_2$ or xylene. M.p. 295°.

Bayer, D.R.P., 107,721, (*Chem. Zentr.*, 1900, I, 1176).

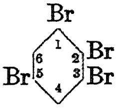
(ii) Yellow needles from CS_2. M.p. above 370°. Sublimes. NaOH fusion ⟶ alizarin.

Hammerschlag, *Ber.*, 1877, **10**, 1213.

(iii) Yellow leaflets from toluene, $CHCl_3$ or CS_2. M.p. 295–300°.

Diehl, *Ber.*, 1878, **11**, 182.

1 : 2 : 3 : 5-Tetrabromobenzene



$C_6H_2Br_4$ MW, 394

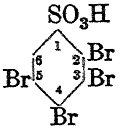
Needles from EtOH. M.p. 98·5°. B.p. 329°. Sol. Et_2O, C_6H_6, CS_2. Mod. sol. EtOH.

v. Richter, *Ber.*, 1875, **8**, 1426.
Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 9, 54.

1 : 2 : 4 : 5-Tetrabromobenzene.
Cryst. from CS_2. M.p. 180–1°. D^{20} 3·027.

Scheufelen, *Ann.*, 1885, **231**, 187.
Jackson, Gallivan, *Am. Chem. J.*, 1896, **18**, 250.
MacKerrow, *Ber.*, 1891, **24**, 2940.
Zelinsky, *Ber.*, 1901, **34**, 2803.

2 : 3 : 4 : 5-Tetrabromobenzenesulphonic Acid



$C_6H_2O_3Br_4S$ MW, 474

Plates from H_2O. M.p. 168–9°. Sol. H_2O. Mod. sol. EtOH, Et_2O.

Chloride : $C_6HO_2ClBr_4S$. MW, 492·5. Plates from Et_2O. M.p. 120°.

Amide: $C_6H_3O_2NBr_4S$. MW, 473. Prisms from EtOH. Sinters and darkens at 240° (161°).

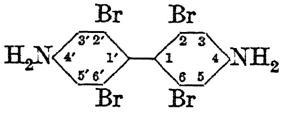
Spiegelberg, *Ann.*, 1879, **197**, 292.
Lenz, *Ann.*, 1876, **181**, 45.

2 : 3 : 4 : 6-Tetrabromobenzenesulphonic Acid.

Needles + $5H_2O$ from H_2O. Sol. EtOH, H_2O.
Chloride: leaflets from Et_2O. M.p. 96·5°.
Amide: needles from EtOH. M.p. about 245° decomp. Spar. sol. H_2O.

Bässmann, *Ann.*, 1878, **191**, 224.
Beckurts, *Ann.*, 1876, **181**, 216.
Knuth, *Ann.*, 1877, **186**, 299.

2 : 6 : 2′ : 6′-Tetrabromobenzidine
(2 : 6 : 2′ : 6′-*Tetrabromo*-4 : 4′-*diaminodiphenyl*)



$C_{12}H_8N_2Br_4$ MW, 500

Cryst. from EtOH.Aq. M.p. 180°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, AcOEt. Spar. sol. ligroin.
4 : 4′-N-*Diacetyl*: prisms from EtOH. M.p. 269–70°.

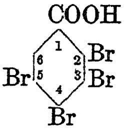
Meyer, Meyer, Taeger, *Ber.*, 1920, **53**, 2045.

3 : 5 : 3′ : 5′-Tetrabromobenzidine
(3 : 5 : 3′ : 5′-*Tetrabromo*-4 : 4′-*diaminodiphenyl*).

Needles from xylene. M.p. 288°. Mod. sol. C_6H_6. Spar. sol. EtOH. Insol. H_2O, Et_2O, acids.
4 : 4′-N-*Diacetyl*: chars at about 340°.
4 : 4′-N-*Tetra-acetyl*: needles from C_6H_6. M.p. about 306°.

van Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.
Schlenk, *Ann.*, 1908, **363**, 335.
Mills, *J. Chem. Soc.*, 1894, **65**, 54.
Claus, Risler, *Ber.*, 1881, **14**, 86.

2 : 3 : 4 : 5-Tetrabromobenzoic Acid



$C_7H_2O_2Br_4$ MW, 438

M.p. 234°. Very sol. EtOH, Me_2CO. Mod. sol. C_6H_6. Spar. sol. pet. ether.
Nitrile: C_7HNBr_4. MW, 419. Needles from EtOH. M.p. 124°.

Bunt, *Rec. trav. chim.*, 1929, **48**, 121.
Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 56.

2 : 3 : 4 : 6-Tetrabromobenzoic Acid.

Needles from C_6H_6. M.p. 179°. Sol. EtOH, Et_2O. Mod. sol. C_6H_6. Spar. sol. H_2O.
Me ester: $C_8H_4O_2Br_4$. MW, 452. Needles from EtOH. M.p. 77°.
Et ester: $C_9H_6O_2Br_4$. MW, 466. M.p. 31°.
Chloride: $C_7HOClBr_4$. MW, 456·5. Prisms from pet. ether. M.p. 58°. Sol. Et_2O, C_6H_6. Spar. sol. H_2O.
Nitrile: needles from EtOH. M.p. 123°. Volatile in steam.

Sudborough, *J. Chem. Soc.*, 1895, **67**, 597.
Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 52.
Sudborough, Karvé, *J. Indian Inst. Sci.*, 1919, **3**, 1.

2 : 3 : 5 : 6-Tetrabromobenzoic Acid.

Yellow plates from EtOH. M.p. 295°. Sol. C_6H_6. Spar. sol. EtOH.
Note.—In the literature this acid is described as of unknown constitution, but since it is different from the other two tetrabromobenzoic acids, it probably possesses the above structure.

Kunckell, Knigge, *Ber.*, 1906, **39**, 195.

Tetrabromo-*p*-benzoquinone.
See Bromanil.

1 : 1 : 2 : 2-Tetrabromobutane

$CH_3{\cdot}CH_2{\cdot}CBr_2{\cdot}CHBr_2$

$C_4H_6Br_4$ MW, 374

Cryst. Sublimes at 200°.

Bruylants, *Ber.*, 1875, **8**, 412.
Dupont, *Compt. rend.*, 1909, **148**, 1523.

1 : 1 : 2 : 3-Tetrabromobutane

$CH_3{\cdot}CHBr{\cdot}CHBr{\cdot}CHBr_2$

$C_4H_6Br_4$ MW, 374

B.p. 148–50°/25 mm.

Muskat, Grimsley, *J. Am. Chem. Soc.*, 1933, **55**, 2145.

1 : 1 : 3 : 3-Tetrabromobutane

$CH_3{\cdot}CBr_2{\cdot}CH_2{\cdot}CHBr_2$

$C_4H_6Br_4$ MW, 374

Fuming liq. D_4^{16} 2·7.

Mereshkowski, *Chem. Zentr.*, 1914, I, 2160.

1 : 2 : 2 : 3-Tetrabromobutane

$CH_3{\cdot}CHBr{\cdot}CBr_2{\cdot}CH_2Br$

$C_4H_6Br_4$ MW, 374

M.p. − 2°. B.p. 97·5°/7 mm. D_4^{20} 2·510. n^{20} 1·6070.

Hurd, Meinert, *J. Am. Chem. Soc.*, 1931, **53**, 293.

1 : 2 : 2 : 4-Tetrabromobutane

$BrCH_2{\cdot}CH_2{\cdot}CBr_2{\cdot}CH_2Br$

$C_4H_6Br_4$ MW, 374

Cryst. from pet. ether. M.p. 72–3°.

Johnson, *J. Chem. Soc.*, 1946, 1009.

1 : 2 : 3 : 4-Tetrabromobutane (*Butadiene tetrabromide, erythrene tetrabromide*)

$BrCH_2{\cdot}CHBr{\cdot}CHBr{\cdot}CH_2Br$

$C_4H_6Br_4$ MW, 374

Exists in two isomeric forms.

(i) Needles from ligroin. M.p. 118–19°. B.p. 180–1°/60 mm. Spar. sol. ligroin. Volatile in steam.

(ii) Leaflets from pet. ether. M.p. 40–1°. Sol. EtOH, Et_2O, ligroin.

Perkin, Simonsen, *J. Chem. Soc.*, 1905, **87**, 857.
Ciamician, Magnaghi, *Ber.*, 1886, **19**, 570; 1887, **20**, 3064.
Jacobson, *J. Am. Chem. Soc.*, 1932, **54**, 1545.

2 : 2 : 3 : 3-Tetrabromobutane

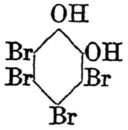
$CH_3{\cdot}CBr_2{\cdot}CBr_2{\cdot}CH_3$

$C_4H_6Br_4$ MW, 374

Cryst. from ligroin. M.p. 243°. Sol. ligroin. Insol. EtOH. Dimorphous.

Faworski, *J. prakt. Chem.*, 1890, **42**, 144.
Wislicenus, Schmidt, *Ann.*, 1900, **313**, 225.

Tetrabromocatechol



$C_6H_2O_2Br_4$ MW, 426

Needles from EtOH. M.p. 192–3°. Sol. EtOH, C_6H_6. Spar. sol. Me_2CO. $FeCl_3 \longrightarrow$ dark blue col.

Mono-Me ether: tetrabromoguaiacol. $C_7H_4O_2Br_4$. MW, 440. Prisms from EtOH or $CHCl_3$. M.p. 162–3°. Sol. Et_2O, Me_2CO. Mod. sol. EtOH, $CHCl_3$. Spar. sol. AcOH. Insol. H_2O.

Di-Me ether: tetrabromoveratrol. $C_8H_6O_2Br_4$. MW, 454. Cryst. from CCl_4. M.p. 151–2° (118–20°).

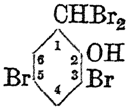
Methylene ether: tetrabromomethylenedioxybenzene. $C_7H_2O_2Br_4$. MW, 438. Needles from EtOH. M.p. 208–9°.

Diacetyl: plates from C_6H_6. M.p. 215–16°.

Dibenzoyl: plates from C_6H_6–ligroin. M.p. 197–8°.

Frejka, Sefránek, *Chem. Zentr.*, 1936, I, 2338.
Jackson, Russe, *Am. Chem. J.*, 1906, **35**, 169, 178.
Zincke, *Ber.*, 1887, **20**, 1778.
Stenhouse, *Ann.*, 1875, **177**, 187.
Zetzsche, Sukiennik, *Helv. Chim. Acta*, 1927, **10**, 101.
Wegmann, Dahn, *Helv. Chim. Acta*, 1946, **29**, 415.

ω : ω : 3 : 5-Tetrabromo-*o*-cresol (*3 : 5-Dibromo-*o*-hydroxybenzylidene bromide*)



$C_7H_4OBr_4$ MW, 424

Needles from pet. ether. M.p. 99°.

Acetyl: needles from pet. ether. M.p. 113–14°.

Lindemann, Forth, *Ann.*, 1924, **435**, 226.

3 : 4 : 5 : 6-Tetrabromo-*o*-cresol.

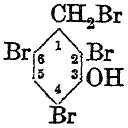
Needles from $CHCl_3$ or AcOH. M.p. 208°. Sol. Et_2O. Mod. sol. $CHCl_3$, C_6H_6. Spar. sol. AcOH, ligroin.

Me ether: $C_8H_6OBr_4$. MW, 438. Needles from AcOH. M.p. 140·5°.

Acetyl: needles from EtOH.Aq. M.p. 154°.

Bonneaud, *Bull. soc. chim.*, 1910, **7**, 779.
Bodroux, *Compt. rend.*, 1898, **126**, 1283.
Zincke, Hedenström, *Ann.*, 1906, **350**, 276.
Anselmino, *Ber.*, 1902, **35**, 150.
Treacy, U.S.P. 2,319,960, (*Chem. Abstracts*, 1943, **37**, 6280).

ω : 2 : 4 : 6-Tetrabromo-*m*-cresol (*2 : 4 : 6-Tribromo-*m*-hydroxybenzyl bromide*)



$C_7H_4OBr_4$ MW, 424

Needles from AcOH. M.p. 149°. Spar. sol. ligroin, pet. ether.

Acetyl: needles from AcOH. M.p. 104°.

Auwers, Richter, *Ber.*, 1899, **32**, 3382.

2 : 4 : 5 : 6-Tetrabromo-*m*-cresol.

Cryst. from AcOH. M.p. 194°.

Me ether: $C_8H_6OBr_4$. MW, 438. Needles from EtOH or AcOH. M.p. 145–6°.

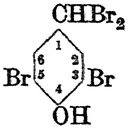
Et ether: $C_9H_8OBr_4$. MW, 452. Needles from Et_2O. M.p. 108°.

Acetyl: needles from ligroin. M.p. 165–6°.

Benzoyl: plates from AcOH. M.p. 153–4°.

Bureš, Balada, *Chem. Abstracts*, 1928, **22**, 3643.
Bonneaud, *Bull. soc. chim.*, 1910, **7**, 780.
Anselmino, *Ber.*, 1902, **35**, 150.
Auwers, Burrows, *Ber.*, 1899, **32**, 3042.

ω : ω : 3 : 5-Tetrabromo-*p*-cresol (*3 : 5-Dibromo-*p*-hydroxybenzylidene bromide*)



$C_7H_4OBr_4$ MW, 424

Needles from pet. ether. M.p. 98–101·5°. Sol. Et_2O, C_6H_6.

Me ether: $C_8H_6OBr_4$. MW, 438. Leaflets from ligroin. M.p. 60–4°.

Acetyl: leaflets from EtOH. M.p. 80°.

Lindemann, *Ann.*, 1923, **431**, 285.

ω : 2 : 3 : 5-Tetrabromo-*p*-cresol (2 : 3 : 5-*Tribromo*-p-*hydroxybenzyl bromide*).

Needles from ligroin. M.p. 122°. Sol. Et_2O, AcOH, C_6H_6. Mod. sol. ligroin.

Acetyl: needles from pet. ether. M.p. 116°.

Zincke, Wiederhold, *Ann.*, 1902, **320**, 210.

Auwers, Strecker, *Ann.*, 1904, **334**, 330.

2 : 3 : 5 : 6-Tetrabromo-*p*-cresol.

Needles from EtOH. M.p. 198–9°. Sol. Et_2O, C_6H_6, hot AcOH. Mod. sol. EtOH. Spar. sol. ligroin.

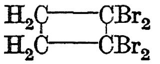
Acetyl: needles from AcOH. M.p. 156°.

Bonneaud, *Bull. soc. chim.*, 1910, **7**, 780.

Zincke, Wiederhold, *Ann.*, 1902, **320**, 207.

Zincke, Buff, *Ann.*, 1905, **341**, 327.

1 : 1 : 2 : 2-Tetrabromocyclobutane



$C_4H_4Br_4$ MW, 372

Prisms from pet. ether. M.p. 126°. Sol. MeOH, EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6.

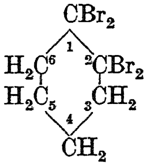
Willstätter, Bruce, *Ber.*, 1907, **40**, 3997.

1 : 2 : 3 : 4-Tetrabromocyclobutane.

Nearly colourless liq. B.p. 110°/1·5 mm. D_4^{20} 2·5673. n_D^{20} 1·6303.

Reppe *et al.*, *Ann.*, 1948, **560**, 86.

1 : 1 : 2 : 2-Tetrabromocyclohexane



$C_6H_8Br_4$ MW, 400

Cryst. from EtOH. M.p. 188–90°.

Domnin, *J. Gen. Chem. U.S.S.R.*, 1946, **16**, 1729, (*Chem. Abstracts*, 1947, **41**, 5861).

1 : 2 : 3 : 4-Tetrabromocyclohexane.

Exists in two forms.

(i) Cryst. from Et_2O. M.p. 87–8°. NaOEt → bromobenzene.

(ii) Cryst. from MeOH. M.p. 155–6°.

Hofmann, Damm, *Chem. Zentr.*, 1926, I, 2343.

Harries, *Ber.*, 1912, **45**, 813.

Bodroux, Taboury, *Compt. rend.*, 1912, **154**, 1514.

Zelinsky, Gorsky, *Ber.*, 1908, **41**, 2483.

1 : 2 : 4 : 5-Tetrabromocyclohexane.

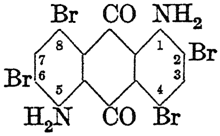
Cryst. from $CHCl_3$. M.p. 184–5°.

Baeyer, *Ann.*, 1894, **278**, 96.

Zelinsky, Gorsky, *Ber.*, 1908, **41**, 2481.

Wibaut, Haak, *Rec. trav. chim.*, 1948, **67**, 95.

2 : 4 : 6 : 8-Tetrabromo-1 : 5-diamino-anthraquinone



$C_{14}H_6O_2N_2Br_4$ MW, 554

Red needles from $PhNO_2$. M.p. about 340°.

Scholl *et al.*, *Ann.*, 1932, **494**, 221.

Mangini, Nicolini, *Chem. Abstracts*, 1946, **40**, 7634.

2 : 4 : 5 : 7-Tetrabromo-1 : 8-diamino-anthraquinone.

Red needles. M.p. above 350°.

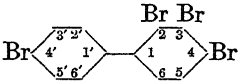
Mangini, Nicolini, *Chem. Abstracts*, 1946, **40**, 7634.

1 : 3 : 5 : 7-Tetrabromo-2 : 6-diamino-anthraquinone.

Yellowish-brown needles from $PhNO_2$. Does not melt below 360°.

Badische, D.R.P., 261,270, (*Chem. Zentr.*, 1913, II, 194).

2 : 3 : 4 : 4′-Tetrabromodiphenyl



$C_{12}H_6Br_4$ MW, 470

Cryst. M.p. 127°.

Bellavita, *Chem. Abstracts*, 1937, **31**, 3902.

2 : 4 : 5 : 4′-Tetrabromodiphenyl.

Cryst. M.p. 135°.

Bellavita, *Chem. Abstracts*, 1937, **31**. 3902.

2 : 4 : 6 : 4′-Tetrabromodiphenyl.

Cryst. from EtOH. M.p. 105–6°.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3489.

2 : 6 : 2′ : 6′-Tetrabromodiphenyl.

Needles from EtOH. M.p. 215°. Mod. sol. common org. solvents.

Meyer, Meyer, Taeger, *Ber.*, 1920, **53**, 2050.

3 : 4 : 3′ : 4′-Tetrabromodiphenyl.

Cryst. from AcOH. M.p. 169°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

3 : 5 : 3′ : 5′-Tetrabromodiphenyl.

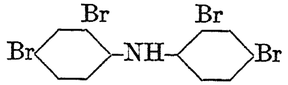
Cryst. from EtOH. M.p. 186°. Sol. Et_2O, C_6H_6. Mod. sol. EtOH.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

Jacobson, *Ann.*, 1909, **367**, 347.

Bellavita, *Chem. Abstracts*, 1937, **31**, 3902.

2 : 4 : 2′ : 4′-Tetrabromodiphenylamine



$C_{12}H_7NBr_4$ MW, 485

Needles or prisms. M.p. 182°. Sol. C_6H_6. Spar. sol. EtOH. Insol. H_2O.

N-*Me* : $C_{13}H_9NBr_4$. MW, 499 Prisms. M.p. 142°. Sol. C_6H_6. Mod. sol. EtOH.

Fries, *Ann.*, 1906, **346**, 213.
Hofmann, *Ann.*, 1864, **132**, 166.

***sym*.-Tetrabromoethane** (*Acetylene tetrabromide*)

$CHBr_2{\cdot}CHBr_2$

$C_2H_2Br_4$ MW, 346

Liq. B.p. 151°/54 mm., 125°/15 mm., 114°/2 mm. D_4^{20} 2·96725. n_D^{20} 1·637951. Zn + EtOH ⟶ *sym*.-dibromoethylene.

O'Meara, Clemmer, *Chem. Abstracts*, 1929, **23**, 1598.
Lespieau, *Compt. rend.*, 1919, **169**, 31.
Reboul, *Ann.*, 1862, **124**, 269.
Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 498.

***unsym*.-Tetrabromoethane**

$BrCH_2{\cdot}CBr_3$

$C_2H_2Br_4$ MW, 346

Liq. B.p. 112·5°/18 mm. D_4^{20} 2·87482. n_D^{20} 1·627721.

Kaufmann, *Ber.*, 1922, **55**, 258.
Lennox, *Ann.*, 1862, **122**, 124.
Reboul, *Ann.*, 1862, **124**, 270.

Tetrabromoethylene (*Perbromoethylene*)

$Br_2C{:}CBr_2$

C_2Br_4 MW, 344

Needles from EtOH. M.p. 56·5°. B.p. 226–7°, 100°/15 mm. Sublimes. Volatile in steam.

Nekrassow, *Ber.*, 1927, **60**, 1758.
Biltz, *Ber.*, 1902, **35**, 1530.
Anschütz, *Ber.*, 1879, **12**, 2073.

Tetrabromoguaiacol.

See under Tetrabromocatechol.

1 : 2 : 2 : 3-Tetrabromohexane

$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CHBr{\cdot}CBr_2{\cdot}CH_2Br$

$C_6H_{10}Br_4$ MW, 402

B.p. 130°/3 mm. D^{15} 2·1873. n_D^{15} 1·5850.

Bouis, *Ann. chim.*, 1928, **9**, 402.

1 : 2 : 3 : 4-Tetrabromohexane (1 : 3-*Hexadiene tetrabromide*)

$CH_3{\cdot}CH_2{\cdot}CHBr{\cdot}CHBr{\cdot}CHBr{\cdot}CH_2Br$

$C_6H_{10}Br_4$ MW, 402

Prisms. M.p. 91–2°.

Fournier, *Bull. soc. chim.*, 1896, **15**, 403.
Prévost, *Ann. chim.*, 1928, **10**, 176.

1 : 2 : 4 : 5-Tetrabromohexane

$CH_3{\cdot}CHBr{\cdot}CHBr{\cdot}CH_2{\cdot}CHBr{\cdot}CH_2Br$

$C_6H_{10}Br_4$ MW, 402

Exists in two forms.

(i) Plates from $CHCl_3$. M.p. 63–4°. Sol. Et_2O, $CHCl_3$, C_6H_6.

(ii) Liq. F.p. below −50°.

Griner, *Ann. chim. phys.*, 1892, **26**, 336.

1 : 2 : 5 : 6-Tetrabromohexane (*Diallyl tetrabromide*)

$BrCH_2{\cdot}CHBr{\cdot}CH_2{\cdot}CH_2{\cdot}CHBr{\cdot}CH_2Br$

$C_6H_{10}Br_4$ MW, 402

Exists in two stereoisomeric forms.

(i) Rhombic cryst. M.p. 64–5°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Mod. sol. ligroin.

(ii) Cryst. M.p. 53–4°.

Griner, *Ann. chim. phys.*, 1892, **26**, 325.
Ciamician, Anderlini, *Ber.*, 1889, **22**, 2498.

2 : 3 : 4 : 5-Tetrabromohexane

$CH_3{\cdot}CHBr{\cdot}CHBr{\cdot}CHBr{\cdot}CHBr{\cdot}CH_3$

$C_6H_{10}Br_4$ MW, 402

Prisms from $CHCl_3$. M.p. 182°.

Prévost, *Ann. chim.*, 1928, **10**, 113.
Paul, Tchelitcheff, *Compt. Rend.*, 1946, **223**, 1138.

Note.—Two other tetrabromohexanes of unknown constitution have been described :

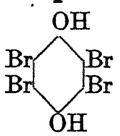
(i) Plates from AcOH. M.p. 162–3°.

Merling, *Ann.*, 1891, **264**, 346.

(ii) M.p. 163–5°.

Mouneyrat, *Ann. chim. phys.*, 1900, **20**, 569.

Tetrabromohydroquinone

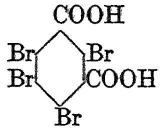


$C_6H_2O_2Br_4$ MW, 426

Prisms from EtOH–Et_2O. M.p. 244°. D^{21} 3·023. Sol. EtOH, Et_2O. Insol. H_2O. $FeCl_3$ in AcOH ⟶ bromanil.

Sarauw, *Ann.*, 1881, **209**, 125.
Bodroux, *Compt. rend.*, 1898, **126**, 1285.
Stenhouse, *Ann.*, 1854, **91**, 310.

Tetrabromoisophthalic Acid



$C_8H_2O_4Br_4$ MW, 482

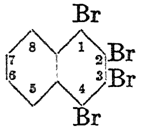
Needles from H_2O. M.p. 288–92°. Insol. C_6H_6.

Rupp, *Ber.*, 1896, **29**, 1631.

Tetrabromomethane.
See Carbon tetrabromide.

Tetrabromomethylenedioxybenzene.
See under Tetrabromocatechol.

1 : 2 : 3 : 4-Tetrabromonaphthalene



$C_{10}H_4Br_4$ MW, 444

Buff needles from EtOH. M.p. 196°.

Hodgson, Hathway, *J. Chem. Soc.*, 1944, 22.

1 : 2 : 6 : 8-Tetrabromonaphthalene.
Needles from C_6H_6. Does not melt below 315°.

Dhar, *J. Chem. Soc.*, 1920, **117**, 993.

1 : 3 : 5 : 8-Tetrabromonaphthalene.
Needles from C_6H_6. M.p. 310°.

Dhar, *J. Chem. Soc.*, 1920, **117**, 993.

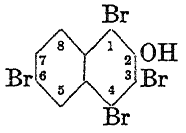
1 : 3 : 6 : 8-Tetrabromonaphthalene.
Needles from C_6H_6. Does not melt below 315°.

Dhar, *J. Chem. Soc.*, 1920, **117**, 993.

1 : 4 : 6 : 7-Tetrabromonaphthalene.
Needles from EtOH. M.p. 175°. Sublimes in plates. Sol. $CHCl_3$, C_6H_6. Very spar. sol. Et_2O.

Guareschi, *Gazz. chim. ital.*, 1886, **16**, 146.

1 : 3 : 4 : 6-Tetrabromo-2-naphthol



$C_{10}H_4OBr_4$ MW, 460

Needles from C_6H_6. M.p. 173–4°. Spar. sol. AcOH. Ox. ⟶ 4-bromophthalic acid.

Me ether: $C_{11}H_6OBr_4$. MW, 474. Needles from pet. ether. M.p. 149°.

Acetyl: needles from C_6H_6–AcOH. M.p. 192°.

Franzen, Stäuble, *J. prakt. Chem.*, 1922, **103**, 377.

Armstrong, Rossiter, *Chem. News*, 1891, **63**, 295.

1 : 3 : 5 : 6-Tetrabromo-2-naphthol.
Needles from $CHCl_3$ or AcOH. M.p. 186°. Sol. C_6H_6. Spar. sol. EtOH, pet. ether.

Acetyl: needles from Me_2CO. M.p. 156°.

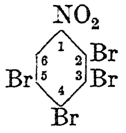
Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 280.

1 : 3 : 6 : 7-Tetrabromo-2-naphthol.
Needles from AcOH or C_6H_6. M.p. 174°. Sol. Et_2O, Me_2CO.

Acetyl: needles from EtOH or AcOH. M.p. 221°.

Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 280.

2 : 3 : 4 : 5-Tetrabromonitrobenzene



$C_6HO_2NBr_4$ MW, 439.

Needles from EtOH. M.p. 107°. Red. ⟶ 2 : 3 : 4 : 5-tetrabromoaniline.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 57.

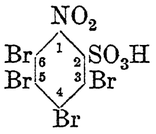
2 : 3 : 4 : 6-Tetrabromonitrobenzene.
Needles from EtOH. M.p. 96°.

v. Richter, *Ber.*, 1875, **8**, 1427.

2 : 3 : 5 : 6-Tetrabromonitrobenzene.
Leaflets from EtOH. M.p. 168°.

Claus, *J. prakt. Chem.*, 1895, **51**, 412.

3 : 4 : 5 : 6-Tetrabromonitrobenzene-2-sulphonic Acid



$C_6HO_5NBr_4S$ MW, 519

Needles + $1H_2O$ from H_2O. M.p. anhyd. 171–3°.

Chloride: $C_6O_4NClBr_4S$. MW, 537·5. Prisms from Et_2O. M.p. 172–3°.

Amide: $C_6H_2O_4N_2Br_4S$. MW, 518. Leaflets. Darkens at 260°, melting with decomp. at higher temp.

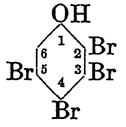
Spiegelberg, *Ann.*, 1878, **197**, 297.

2 : 4 : 5 : 6-Tetrabromonitrobenzene-3-sulphonic Acid.
Needles. Easily sol. H_2O, EtOH. Conc. HCl in H_2SO_4 at 200° ⟶ 2 : 3 : 4 : 6-tetrabromonitrobenzene.

Chloride: plates from Et_2O. M.p. 147·5°.

Langfurth, *Ann.*, 1878, **191**, 202.

2 : 3 : 4 : 5-Tetrabromophenol



$C_6H_2OBr_4$ MW, 410

White needles from EtOH.Aq. M.p. 123°.

Acetyl: white needles from AcOH.Aq. M.p. 110·5°.

Benzoyl: white needles from EtOH.Aq. M.p. 133°.

Farinholt, Stuart, Twiss, *J. Am. Chem. Soc.*, 1940, **62**, 1240.

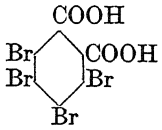
2 : 3 : 4 : 6-Tetrabromophenol
Needles from AcOH. M.p. 111°. Very sol. EtOH. Sublimes.

Me ether: 2 : 3 : 4 : 6-Tetrabromoanisole. $C_7H_4OBr_4$. MW, 424. Cryst. from EtOH. M.p. 104°. B.p. 340°.

Acetyl: leaflets from AcOH.Aq. M.p. 104–5°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.
Körner, *Ann.*, 1866, **137**, 210.
Kohn, Krasso, *J. Org. Chem.*, 1946, **11**, 645.

Tetrabromophthalic Acid



$C_8H_2O_4Br_4$ MW, 482

Needles from H_2O. M.p. 266° ⟶ anhydride.

Mono-Me ester: $C_9H_4O_4Br_4$. MW, 496. Cryst. from MeOH.Aq. M.p. 267°. Sol. EtOH, Et_2O, C_6H_6.

Anhydride: $C_8O_3Br_4$. MW, 464. Cryst. from AcOH–xylene. M.p. 279·5–280·5°.

Imide: $C_8HO_2NBr_4$. MW, 463. Yellow cryst. from AcOH. Does not melt below 380°. Spar. sol. C_6H_6, toluene, AcOH.

Anil: plates from AcOH. M.p. 279–80°.

o-*Nitroanil*: needles from AcOH. M.p. 298–298·5°.

m-*Nitroanil*: plates from xylene. M.p. 301·5–303°.

p-*Nitroanil*: needles from xylene. M.p. 331–331·5°.

o-*Tolil*: plates from C_6H_6 or AcOH. M.p. 291–3°.

m-*Tolil*: cryst. from C_6H_6. M.p. 273·5–274·5°.

p-*Tolil*: yellow needles from C_6H_6. M.p. 280–280·5°.

α-*Naphthylimide*: yellow needles from C_6H_6. M.p. 309–309·5°.

β-*Naphthylimide*: greenish-yellow plates. M.p. 305·5–308°.

Pratt, Young, *J. Am. Chem. Soc.*, 1918, **40**, 1416.
Hofmann, *Monatsh.*, 1915, **36**, 818.

1 : 1 : 1 : 2-Tetrabromopropane

$CH_3{\cdot}CHBr{\cdot}CBr_3$

$C_3H_4Br_4$ MW, 360

B.p. 122°/15 mm. D_4^{20} 2·679. n_D^{20} 1·6187.

Bachman, *J. Am. Chem. Soc.*, 1935, **57**, 1090.

1 : 1 : 1 : 3-Tetrabromopropane

$CH_2Br{\cdot}CH_2{\cdot}CBr_3$

$C_3H_4Br_4$ MW, 360

Liq. B.p. 65–8°/0·3 mm. n_D^{20} 1·6090.

Kharasch, Jensen, Urry, *J. Am. Chem. Soc.*, 1946, **68**, 155; 1947, **69**, 1104.

1 : 1 : 2 : 2-Tetrabromopropane (*Allylene tetrabromide*)

$CH_3{\cdot}CBr_2{\cdot}CHBr_2$

$C_3H_4Br_4$ MW, 360

Liq. B.p. 225–30° part. decomp., 105–7°/9 mm. D_4^{20} 2·687. n_D^{20} 1·6166.

Hurd, Meinert, Spence, *J. Am. Chem. Soc.*, 1930, **52**, 1142.
Oppenheim, *Ann.*, 1864, **132**, 124.

1 : 1 : 2 : 3-Tetrabromopropane

$BrCH_2{\cdot}CHBr{\cdot}CHBr_2$

$C_3H_4Br_4$ MW, 360

B.p. 179–80°/80 mm., 138–40°/17 mm. D^0 2·76.

Mouneyrat, *Compt. rend.*, 1898, **127**, 276.
Lespieau, *Ann. chim. phys.*, 1897, **11**, 253.

1 : 1 : 3 : 3-Tetrabromopropane

$Br_2CH{\cdot}CH_2{\cdot}CHBr_2$

$C_3H_4Br_4$ MW, 360

B.p. 154–6°/19 mm. D_4^0 2·7405, D_4^{21} 2·702. n_D^{21} 1·6225.

Demjanow, Dojarenko, *Ber.*, 1923, **56**, 2202.

1 : 2 : 2 : 3-Tetrabromopropane (*Allene tetrabromide*)

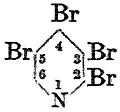
$BrCH_2{\cdot}CBr_2{\cdot}CH_2Br$

$C_3H_4Br_4$ MW, 360

Colourless liq. with odour resembling camphor. M.p. 10–11°. B.p. 215–30° decomp., 169–70°/80 mm., 115·5°/9 mm. D_4^{20} 2·703. n_D^{20} 1·6200. Zn dust in EtOH ⟶ allene.

Gustavson, Demjanow, *J. prakt. Chem.*, 1888, **38**, 204.
Hurd, Meinert, Spence, *J. Am. Chem. Soc.*, 1930, **52**, 1143.

2 : 3 : 4 : 5-Tetrabromopyridine



C_5HNBr_4 MW, 395

Cryst. from EtOH.Aq. M.p. 74·5–75·5°.

Hertog, *Rec. trav. chim.*, 1945, **64**, 101.

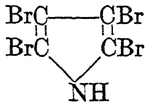
2 : 3 : 4 : 6-Tetrabromopyridine. Cryst. from EtOH. M.p. 105–6°.

Wibaut, Bickel, *Rec. trav. chim.*, 1939, **58**, 996.

2 : 3 : 5 : 6-Tetrabromopyridine. Needles from 50% AcOH. M.p. 103·5–104°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.
Wibaut, Wagtendonk, *Rec. trav. chim.*, 1941, **60**, 24.

2 : 3 : 4 : 5-Tetrabromopyrrole



C_4HNBr_4 MW, 383

Needles from EtOH. Does not melt below 250°. Sol. EtOH. Spar. sol. H_2O. KI in EtOH ⟶ 2 : 3 : 4 : 5-tetraiodopyrrole.

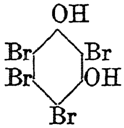
N-*Me*: $C_5H_3NBr_4$. MW, 397. Needles from pet. ether. M.p. 154–5° ⟶ a blue liq.

N-*Et*: $C_6H_5NBr_4$. MW, 411. Needles from EtOH. M.p. 90° (83°). Spar. sol. EtOH. Insol. H_2O.

Plancher, Soncini, *Gazz. chim. ital.*, 1902, **32**, ii, 465.
Zanetti, *Ber.*, 1889, **22**, 2515.
de Varda, *Ber.*, 1888, **21**, 2871.

Tetrabromoquinone.
See Bromanil.

Tetrabromoresorcinol



$C_6H_2O_2Br_4$ MW, 426

Needles from EtOH.Aq. M.p. 167° (163°). Sol. Et_2O, $CHCl_3$, hot EtOH. Spar. sol. H_2O.

Diacetyl: m.p. 114° (169°).

Claassen, *Ber.*, 1878, **11**, 1440.
Benedikt, *Monatsh.*, 1880, **1**, 366.

5 : 5 : 6 : 6-Tetrabromostearic Acid (*Taric acid tetrabromide*)

$CH_3\cdot[CH_2]_{10}\cdot CBr_2\cdot CBr_2\cdot[CH_2]_4\cdot COOH$

$C_{18}H_{32}O_2Br_4$ MW, 600

M.p. 125°.

Arnaud, *Compt. rend.*, 1902, **134**, 842; *Bull. soc. chim.*, 1892, **7**, 234.

8 : 8 : 9 : 9-Tetrabromostearic Acid (*Stearolic acid tetrabromide*)

$CH_3\cdot[CH_2]_7\cdot CBr_2\cdot CBr_2\cdot[CH_2]_7\cdot COOH$

$C_{18}H_{32}O_2Br_4$ MW, 600

Cryst. from EtOH. M.p. 70°.

Overbeck, *Ann.*, 1866, **140**, 56.

8 : 9 : 11 : 12-Tetrabromostearic Acid (*Linoleic acid tetrabromide*)

$CH_3\cdot[CH_2]_4\cdot[CHBr]_2\cdot CH_2\cdot[CHBr]_2\cdot[CH_2]_7\cdot COOH$

$C_{18}H_{32}O_2Br_4$ MW, 600

Cryst. from Et_2O–pet. ether. M.p. 114·7–115·2°. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Insol. H_2O. Sn + HCl in EtOH ⟶ linoleic acid.

K salt: cryst. from EtOH. M.p. 171° (154·7–158·8°).

Na salt: cryst. from EtOH. M.p. 194·2–201·1°.

Ca salt: cryst. from EtOH. M.p. 208·7–213·4°.

Sr salt: cryst. from EtOH. M.p. 200·4–206°.

Ba salt: cryst. from EtOH. M.p. 196·3–206·5.°

S-*Benzylisothiouronium salt*: cryst. from EtOH. M.p. 129–130°.

Me ester: $C_{19}H_{34}O_2Br_4$. MW, 614. M.p. 63° (50–6°).

Et ester: $C_{20}H_{36}O_2Br_4$. MW, 628. Needles. M.p. 63° (58–58·5°).

Propyl ester: $C_{21}H_{38}O_2Br_4$. MW, 642. Cryst. from MeOH. M.p. 45–50°.

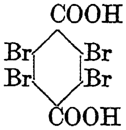
Isopropyl ester: m.p. 50–2°.

Allyl ester: $C_{21}H_{36}O_2Br_4$. MW, 640. M.p. 72–80°.

p-*Phenylphenacyl ester*: cryst. from Me_2CO. M.p. 107–8°.

Smit, *Rec. trav. chim.*, 1930, **49**, 539.
Santos, West, *Chem. Abstracts*, 1928, **22**, 761.
Oreta, West, *Chem. Abstracts*, 1927, **21**, 3889.
Hazura, *Monatsh.*, 1887, **8**, 149.
Rollet, *Z. physiol. Chem.*, 1909, **62**, 414.
Palmer, Wright, *Ind. Eng. Chem.*, 1914, **6**, 822.
McCutcheon, *Organic Syntheses*, 1942, XXII, 76.

Tetrabromoterephthalic Acid



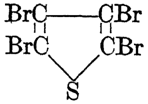
$C_8H_2O_4Br_4$ MW, 482

Needles from H_2O. M.p. about 300° decomp. Very spar. sol. EtOH, Et_2O, AcOH. Insol. C_6H_6, cold H_2O.

Rupp, *Ber.*, 1896, **29**, 1626.

Tetrabromotetramethylmethane.
See Pentaerythrityl tetrabromide.

Tetrabromothiophene



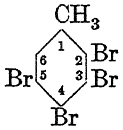
C_4Br_4S MW, 400

Needles from EtOH. M.p. 117–18°. B.p. 326°, 170–3°/13 mm. Fuming HNO_3 ⟶ dibromomaleic acid.

Steinkopf, Jacob, Penz, *Ann.*, 1934, **512**, 149.

α : 2 : 4 : 6-Tetrabromotoluene.
See 2 : 4 : 6-Tribromobenzyl bromide.

2 : 3 : 4 : 5-Tetrabromotoluene



$C_7H_4Br_4$ MW, 408

Needles. M.p. 111–111·5°.

Nevile, Winther, *Ber.*, 1880, **13**, 976.
Schmid, *Helv. Chim. Acta*, 1946, **29**, 1151.
Qvist, *Chem. Abstracts*, 1947, **41**, 2398.

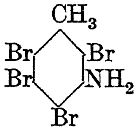
2 : 3 : 4 : 6-Tetrabromotoluene.
M. p. 105–8°.

Nevile, Winther, *Ber.*, 1880, **13**, 975.

2 : 3 : 5 : 6-Tetrabromotoluene.
Needles. M.p. 116–17°. Spar. sol. EtOH.

Nevile, Winther, *Ber.*, 1880, **13**, 975.

2 : 4 : 5 : 6-Tetrabromo-*m*-toluidine

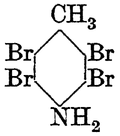


$C_7H_5NBr_4$ MW, 423

M.p. 223–4°. Mod. sol. EtOH.

Nevile, Winther, *Ber.*, 1880, **13**, 975.
Qvist *et al.*, *Chem. Abstracts*, 1942, **36**, 5788.

2 : 3 : 5 : 6-Tetrabromo-*p*-toluidine



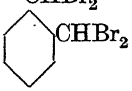
$C_7H_5NBr_4$ MW, 423

Needles. M.p. 226–7°.

Nevile, Winther, *Ber.*, 1881, **14**, 418.
Scheufelen, *Ann.*, 1885, **231**, 179.

Tetrabromoveratrol.
See under Tetrabromocatechol.

ω : ω : ω′ : ω′-**Tetrabromo-*o*-xylene**

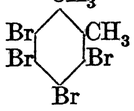


$C_8H_6Br_4$ MW, 422

Cryst. from EtOH. M.p. 116°. Sol. $CHCl_3$. Somewhat difficultly sol. EtOH. Insol. ligroin. K oxalate in EtOH.Aq. ⟶ *o*-phthalaldehyde.

Gabriel, Müller, *Ber.*, 1895, **28**, 1830.
Thiele, Günther, *Ann.*, 1906, **347**, 107.

3 : 4 : 5 : 6-Tetrabromo-*o*-xylene

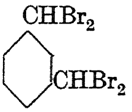


$C_8H_6Br_4$ MW, 422

Needles from C_6H_6. M.p. 262° (254–5°). B.p. 374–5°. Sol. hot C_6H_6. Very spar. sol. hot EtOH. Dil. HNO_3 + Br at 170° ⟶ tetrabromophthalic acid.

Blümlein, *Ber.*, 1884, **17**, 2492.
Klages, Sommer, *Ber.*, 1906, **39**, 2312.
Hennion, Anderson, *J. Am. Chem. Soc.*, 1946, **68**, 425.

ω : ω : ω′ : ω′-**Tetrabromo-*m*-xylene**

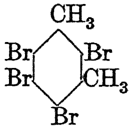


$C_8H_6Br_4$ MW, 422

Needles from EtOH. M.p. 107°. Sol. $CHCl_3$, AcOH, C_6H_6, ligroin. K oxalate in EtOH ⟶ isophthalaldehyde.

Thiele, Günther, *Ann.*, 1906, **347**, 109.

2 : 4 : 5 : 6-Tetrabromo-*m*-xylene

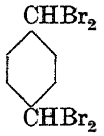


$C_8H_6Br_4$ MW, 422

M.p. 247° (241°). Sol. C_6H_6. Insol. EtOH.

Datta, Chatterjee, *J. Am. Chem. Soc.*, 1916, **38**, 2550.
Hennion, Anderson, *J. Am. Chem. Soc.*, 1946, **68**, 425.

ω : ω : ω′ : ω′-**Tetrabromo-*p*-xylene**

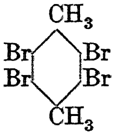


$C_8H_6Br_4$ MW, 422

Prisms from $CHCl_3$. M.p. 169°. Sol. C_6H_6. Spar. sol. EtOH. Hot. conc. H_2SO_4 ⟶ terephthalaldehyde + terephthalaldehydic acid.

Hönig, *Monatsh.*, 1888, **9**, 1150.
Thiele, Günther, *Ann.*, 1906, **347**, 110.
Snell, Weissberger, *Organic Syntheses*, 1940, XX, 92.

2 : 3 : 5 : 6-Tetrabromo-*p*-xylene



$C_8H_6Br_4$ MW, 422

Needles from toluene. M.p. 256–7° (253°). B.p. 355°. Very spar. sol. hot H_2O.

Zelinsky, Lepeschkin, *Chem. Zentr.*, 1913, II, 2126.
Jacobsen, *Ber.*, 1885, **18**, 359.
Hennion, Anderson, *J. Am. Chem. Soc.*, 1946, **68**, 425.

Tetrabutylammonium iodide.
See under Tributylamine.

Tetracarboxytetramethylmethane.
See Methane-tetracetic Acid.

Tetrachloroacetanilide.
See under Tetrachloroaniline.

1 : 1 : 1 : 3-Tetrachloroacetone

$CH_2Cl{\cdot}CO{\cdot}CCl_3$

$C_3H_2OCl_4$ MW, 196

Liq. with acrid odour which affects mucous membrane. B.p. 183°. D_4^{15} 1·624. n_D^{18} 1·497. Forms hydrate. $NH_3 \longrightarrow CHCl_3$ and chloroacetamide.

Hydrate: $C_3H_2OCl_4,4H_2O$. MW, 268. Prisms. M.p. 46°.

Brochet, *Compt. rend.*, 1894, **119**, 1271.

1 : 1 : 3 : 3-Tetrachloroacetone

$CHCl_2{\cdot}CO{\cdot}CHCl_2$

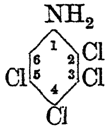
$C_3H_2OCl_4$ MW, 196

Liq. with strong acrid odour. B.p. 180–2°/718 mm. Forms cryst. hydrate. Forms bisulphite comp. Zn + $H_2SO_4 \longrightarrow$ acetone.

Hydrate: $C_3H_2OCl_4,4H_2O$. MW, 268. Plates. M.p. 48–9°.

Zincke, Kegel, *Ber.*, 1889, **22**, 1478.

2 : 3 : 4 : 5-Tetrachloroaniline



$C_6H_3NCl_4$ MW, 231

Needles from EtOH. M.p. 118–20°. Very sol. EtOH, Et_2O, AcOH, C_6H_6.

N-*Acetyl*: 2 : 3 : 4 : 5-tetrachloroacetanilide. $C_8H_5ONCl_4$. MW, 273. Cryst. M.p. 160–2°. Very sol. EtOH, Et_2O, AcOH.

Grandmougin, Seyder, *Ber.*, 1914, **47**, 2369.

Beilstein, Kurbatow, *Ann.*, 1879, **196**, 237.

2 : 3 : 5 : 6-Tetrachloroaniline.

Colourless needles from EtOH. M.p. 107–8°. Very sol. EtOH, CS_2, ligroin.

N-*Acetyl*: 2 : 3 : 5 : 6-tetrachloroacetanilide. Colourless needles from EtOH. M.p. 213–14°. Very sol. EtOH. Spar. sol. AcOH.Aq. N-*Me*: m.p. 96–7°. N-*Et*: m.p. 73–4°.

N-*Diacetyl*: colourless needles from EtOH. M.p. 175–6°.

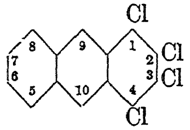
Azo-β-naphthol deriv.: red needles from AcOH. M.p. 212°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3044.

Peters, Rowe, Stead, *J. Chem. Soc.*, 1943, 233.

Tetrachloroanisole.
See under Tetrachlorophenol.

1 : 2 : 3 : 4-Tetrachloroanthracene



$C_{14}H_6Cl_4$ MW, 316

Needles from EtOH–$CHCl_3$. M.p. 148–9°. Very sol. $CHCl_3$, CS_2, C_6H_6. Mod. sol. hot AcOH. Spar. sol. EtOH, Et_2O. Ox. ⟶ 1 : 2 : 3 : 4-tetrachloroanthraquinone.

Kircher, *Ann.*, 1887, **238**, 346.

1 : 3 : 9 : 10-Tetrachloroanthracene.

Yellow needles from AcOH. M.p. 164°. Sol. boiling AcOH. Spar. sol. EtOH. Ox. ⟶ 1 : 3-dichloroanthraquinone.

Höchst, D.R.P., 282,818, (*Chem. Zentr.*, 1915, I, 772).

Meyer, Zahn, *Ann.*, 1913, **396**, 172.

1 : 4 : 5 : 8-Tetrachloroanthracene.

M.p. 285–6° (275°).

Note.—The positions occupied by the chlorine atoms are not definitely established.

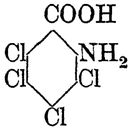
Schilling, *Ber.*, 1913, **46**, 1068.

2 : 3 : 9 : 10-Tetrachloroanthracene.

Yellow needles from C_6H_6. M.p. 240–1°. Spar. sol. ord. org. solvents except hot C_6H_6. Ox. ⟶ 2 : 3-dichloroanthraquinone.

Meyer, Zahn, *Ann.*, 1913, **396**, 177.

Tetrachloroanthranilic Acid



$C_7H_3O_2NCl_4$ MW, 275

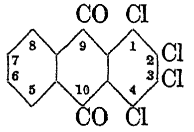
Needles from H_2O or AcOH. M.p. 182°. Very sol. EtOH, Et_2O. Spar. sol. cold AcOH, C_6H_6. Insol. cold H_2O. Forms cryst. spar. sol. Ca, Ba salts. Heat above m.p. ⟶ 2 : 3 : 4 : 5-tetrachloroaniline.

Me ester: $C_8H_5O_2NCl_4$. MW, 289. Needles from MeOH. M.p. 120–1°.

Badische, D.R.P., 220,839, (*Chem. Zentr.*, 1910, I, 1564).

Villiger, Blangey, *Ber.*, 1909, **42**, 3550.

1 : 2 : 3 : 4-Tetrachloroanthraquinone



$C_{14}H_4O_2Cl_4$ MW, 346

Yellow needles. M.p. 191°. Very sol. C_6H_6, $CHCl_3$. Spar. sol. AcOH. Very spar. sol. EtOH, Et_2O. $HNO_3 \longrightarrow$ tetrachlorophthalic acid.

Kircher, *Ann.*, 1887, **238**, 345.

1 : 2 : 5 : 8-Tetrachloroanthraquinone.
Felted needles from AcOH. M.p. 282–3°.

Goldberg, *J. Chem. Soc.*, 1931, 1792.

1 : 2 : 6 : 7-Tetrachloroanthraquinone.
Golden-yellow needles from xylene. M.p. 242°. Sol. cold conc. H_2SO_4.

Barnett, Goodway, Watson, *Ber.*, 1933, **66**, 1885.

1 : 4 : 5 : 8-Tetrachloroanthraquinone.
Yellow needles from AcOH or $PhNO_2$. M.p. 341–2°. Sol. $H_2SO_4 \longrightarrow$ yellow col.

I.C.I., U.S.P., 1,969,044, (*Chem. Abstracts*, 1934, **28**, 6159); B.P. 364,141, (*Chem. Zentr.*, 1933, I, 2174).
Goldberg, *J. Chem. Soc.*, 1931, 1792.
Beard, U.S.P. 2,378,745, (*Chem. Abstracts*, 1946, **40**, 365).

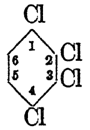
1 : 4 : 6 : 7-Tetrachloroanthraquinone.
Cryst. from $CHCl_3$. M.p. 259–60°.

Allen, Frame, Wilson, *J. Org. Chem.*, 1942, **7**, 65.

2 : 3 : 6 : 7-Tetrachloroanthraquinone.
Pale yellow cryst. from *o*-dichlorobenzene. M.p. 348°. Insol. cold. conc. H_2SO_4.

Barnett, Goodway, Watson, *Ber.*, 1933, **66**, 1885.

1 : 2 : 3 : 4-Tetrachlorobenzene



$C_6H_2Cl_4$ MW, 216

Needles. M.p. 45–6°. B.p. 254°. Very sol. Et_2O, AcOH, CS_2, ligroin. Spar. sol. EtOH.

Cohen, Hartley, *J. Chem. Soc.*, 1905, **87**, 1365.
Nordlander, Cass, *J. Am. Chem. Soc.*, 1947, **69**, 2681.

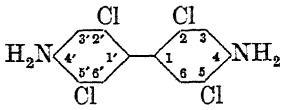
1 : 2 : 3 : 5-Tetrachlorobenzene.
Needles from EtOH. M.p. 51°. B.p. 246°. Very sol. CS_2, ligroin. Sol. C_6H_6. Spar. sol. cold EtOH.

Willgerodt, Wilcke, *Ber.*, 1910, **43**, 2752.

1 : 2 : 4 : 5-Tetrachlorobenzene.
Needles from EtOH, Et_2O, C_6H_6, C_6H_6–EtOH or CS_2. M.p. 139·5–140·5°. B.p. 240°. Sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. boiling EtOH. Insol. cold EtOH.

Qvist, *Chem. Zentr.*, 1934, II, 595.
Dow, U.S.P., 1,934,675, (*Chem. Zentr.*, 1934, I, 1390).
Pollak, Wienerberger, *Monatsh.*, 1914, **35**, 1472.

2 : 5 : 2′ : 5′-Tetrachlorobenzidine



$C_{12}H_8N_2Cl_4$ MW, 322

Orange flakes from EtOH. M.p. 137·5°.
B,HCl: m.p. 230°.
N : N′-*Diacetyl*: cryst. from AcOH. M.p. 297°.
N : N′-*Dibenzoyl*: colourless needles from AcOH. M.p. 250–2°.

Govindachari, Rajagopalan, *Chem. Abstracts*, 1947, **41**, 4046.
Govindachari, Rajagopalan, Venkatakrishna, *Chem. Abstracts*, 1949, **43**, 3729.

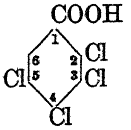
2 : 6 : 2′ : 6′-Tetrachlorobenzidine.
Cryst. M.p. 212°.
4 : 4′-N-*Diacetyl*: m.p. 312°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

3 : 5 : 3′ : 5′-Tetrachlorobenzidine.
Needles from EtOH–toluene. M.p. 226°. Very sol hot C_6H_6. Spar. sol. EtOH. Insol. Et_2O, ligroin.
4 : 4′-N-*Diacetyl*: m.p. 350°.
Tetra-acetyl: needles from AcOH. M.p. 265–6°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.
Schlenk, *Ann.*, 1908, **363**, 334.

2 : 3 : 4 : 5-Tetrachlorobenzoic Acid



$C_7H_2O_2Cl_4$ MW, 260

Cryst. from Me_2CO–H_2O. M.p. 194–5°. Sol. EtOH, Et_2O. Spar. sol. H_2O.
Et ester: $C_9H_6O_2Cl_4$. MW, 288. Needles from EtOH. M.p. 34·5°.
Chloride: m.p. 37–8°. B.p. 99–101°/0·2 mm.
Amide: cryst. from Me_2CO–EtOH. M.p. 207–8°.
Nitrile: C_7HNCl_4. MW, 241. Needles. M.p. 84°.
Anilide: cryst. from EtOH. M.p. 197–197·5°.

Tust, *Ber.*, 1887, **20**, 2439.
Nordlander, Cass, *J. Am. Chem. Soc.*, 1947, **69**, 2680.

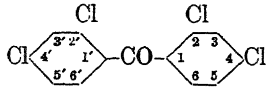
2 : 3 : 4 : 6-Tetrachlorobenzoic Acid.
Nitrile: needles. M.p. 81°.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 48, 66.

2 : 3 : 5 : 6-Tetrachlorobenzoic Acid.
Nitrile: needles. M.p. 72°.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 48, 66.

2 : 4 : 2′ : 4′-Tetrachlorobenzophenone



$C_{13}H_6OCl_4$ MW, 320

Cryst. M.p. 78°. Spar. sol. EtOH.Aq.

Cohen, *Rec. trav. chim.*, 1919, **38**, 116.

2 : 5 : 2′ : 4′-Tetrachlorobenzophenone. Needles from EtOH. M.p. 176°.

Ganzmüller, *J. prakt. Chem.*, 1933, **138**, 312.

2 : 5 : 2′ : 5′-Tetrachlorobenzophenone. Plates from EtOH. M.p. 128°. Very sol. hot EtOH, pet. ether. Spar. sol. cold EtOH.

Norris, Green, *Am. Chem. J.*, 1901, **26**, 498.

3 : 4 : 3′ : 4′-Tetrachlorobenzophenone. Cryst. from EtOH. M.p. 141–2°.

Wilson, Cheng, *J. Org. Chem.*, 1940, **5**, 225.

1 : 1 : 1 : 2-Tetrachlorobutane

$CH_3{\cdot}CH_2{\cdot}CHCl{\cdot}CCl_3$

$C_4H_6Cl_4$ MW, 196

Liq. B.p. 134–5°/742 mm. D^{20}_{25} 1·3932. n^{25}_{D} 1·4920.

Gilman, Abbott, *J. Org. Chem.*, 1943, **8**, 228.

1 : 2 : 2 : 3-Tetrachlorobutane

$CH_3{\cdot}CHCl{\cdot}CCl_2{\cdot}CH_2Cl$

$C_4H_6Cl_4$ MW, 196

Liq. B.p. 85°/10 mm.

Garzarolli-Thurnlackh, *Ann.*, 1882, **213**, 372.

1 : 2 : 3 : 3-Tetrachlorobutane

$CH_3{\cdot}CCl_2{\cdot}CHCl{\cdot}CH_2Cl$

$C_4H_6Cl_4$ MW, 196

Liq. B.p. 90°/32 mm., 55–7°/10 mm. D^{20}_{4} 1·4204. n^{20}_{D} 1·4958.

du Pont, U.S.P., 1,964,720, (*Chem. Zentr.*, 1934, II, 3180).
Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **44**, 1631.

1 : 2 : 3 : 4-Tetrachlorobutane (*Butadiene tetrachloride, erythrene tetrachloride*)

$CH_2Cl{\cdot}CHCl{\cdot}CHCl{\cdot}CH_2Cl$

$C_4H_6Cl_4$ MW, 196

Prisms with camphor-like odour from EtOH. M.p. 73–4°. B.p. 130–40°/50 mm.

Backer, Strating, *Rec. trav. chim.*, 1935, **54**, 55.
Henninger, *Ann. chim. phys.*, 1886, **7**, 229.

2 : 2 : 3 : 3-Tetrachlorobutane

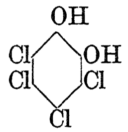
$CH_3{\cdot}CCl_2{\cdot}CCl_2{\cdot}CH_3$

$C_4H_6Cl_4$ MW, 196

M.p. 174°.

McBee, Hausch, *Ind. Eng. Chem.*, 1947, **39**, 418.

Tetrachlorocatechol



$C_6H_2O_2Cl_4$ MW, 248

Cryst. from EtOH.Aq. or C_6H_6: cryst. + $1H_2O$ from AcOH.Aq. M.p. 194–5°, anhyd. 110°. Cryst. + $3H_2O$ from AcOH.Aq., m.p. 94°. Spar. sol. C_6H_6. Ox. ⟶ tetrachloro-*o*-benzoquinone.

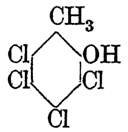
Mono-Me ether: tetrachloroguaiacol. $C_7H_4O_2Cl_4$. MW, 262. Cryst. from hot H_2O. M.p. 185–6°.

Di-Me ether: tetrachloroveratrol. $C_8H_6O_2Cl_4$. MW, 276. Needles from AcOH. M.p. 190°.

Diacetyl: needles from AcOH. M.p. 190°.

Zincke, Küster, *Ber.*, 1888, **21**, 2729.

Tetrachloro-*o*-cresol



$C_7H_4OCl_4$ MW, 246

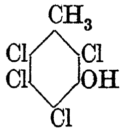
Needles from C_6H_6. M.p. 190°. Very sol. EtOH, Et_2O, AcOH, C_6H_6.

Me ether: $C_8H_6OCl_4$. MW, 260. Needles from MeOH. M.p. 114°.

Acetyl: needles from AcOH. M.p. 136°.

Zincke, Pfaffendorf, *Ann.*, 1912, **394**, 12.

Tetrachloro-*m*-cresol



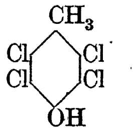
$C_7H_4OCl_4$ MW, 246

Needles from pet. ether. M.p. 189–90°. Sol. ord. org. solvents.

Acetyl: needles from AcOH. M.p. 117°.

Crowther, McCombie, *J. Chem. Soc.*, 1913, **103**, 546.

Tetrachloro-*p*-cresol



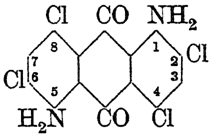
$C_7H_4OCl_4$ MW, 246

Needles from AcOH or C_6H_6–ligroin. M.p. 190°. Very sol. EtOH, AcOH, $CHCl_3$, C_6H_6. Spar. sol. ligroin.

Acetyl· needles from C_6H_6, plates from AcOH.Aq. M.p. 112°.

Zincke, *Ann.*, 1903, **328**, 281.

2 : 4 : 6 : 8 - Tetrachloro - 1 : 5 - diamino-anthraquinone



$C_{14}H_6O_2N_2Cl_4$ MW, 376

Brown needles from $PhNO_2$. Spar. sol. cold $PhNO_2$. Insol. EtOH, cold AcOH. H_2SO_4 ⟶ olive col.

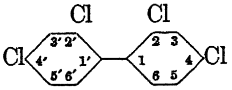
Badische, D.R.P., 158,951, (*Chem. Zentr.*, 1905, I, 842).

2 : 4 : 5 : 7 - Tetrachloro - 1 : 8 - diamino-anthraquinone.

Pale brown plates from $PhNO_2$. Sol. hot $PhNO_2$. Spar. sol. EtOH. H_2SO_4 ⟶ yellow col.

Badische, D.R.P. 158,951, (*Chem. Zentr.*, 1905, I, 842).

2 : 4 : 2′ : 4′-Tetrachlorodiphenyl



$C_{12}H_6Cl_4$ MW, 292

Cryst. from C_6H_6–ligroin. M.p. 83°. Very sol. warm EtOH, C_6H_6. Spar. sol. ligroin.

Ullmann, *Ann.*, 1904, **332**, 55.

2 : 5 : 2′ : 5′-Tetrachlorodiphenyl.

Yellow cryst. from MeOH or pet. ether. M.p. 84–5°.

Meyer, Hofmann, *Monatsh.*, 1917, **38**, 145.

2 : 6 : 2′ : 6′-Tetrachlorodiphenyl.

Plates. M.p. 199°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

3 : 4 : 3′ : 4′-Tetrachlorodiphenyl.

Colourless needles from AcOH. M.p. 171°. B.p. 230°/50 mm. Very sol. EtOH, Et_2O.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

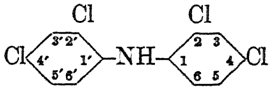
Cain, *J. Chem. Soc.*, 1904, **85**, 7.

3 : 5 : 3′ : 5′-Tetrachlorodiphenyl.

M.p. 162°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

2 : 4 : 2′ : 4′-Tetrachlorodiphenylamine



$C_{12}H_7NCl_4$ MW, 307

Prisms or needles. M.p. 141–2° (134°). Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6.

N-*Me*: $C_{13}H_9NCl_4$. MW, 321. Prisms. M.p. 96–7°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6.

Krollpfeiffer, Wolf, Walbrecht, *Ber.*, 1934, **67**, 908.

Gnehm, *Ber.*, 1875, **8**, 1040.

2 : 3 : 5 : 4′-Tetrachlorodiphenylamine.

Cryst. from EtOH.Aq. M.p. 107–8°. Very sol. EtOH, Et_2O, ligroin.

Jacobson, *Ann.*, 1909, **367**, 339.

1 : 1 : 1 : 2-Tetrachloroethane

$CHCl{\cdot}CCl_3$

$C_2H_2Cl_4$ MW, 168

Liq. B.p. 135·1°. D_0^{25} 1·5424.

I.G., D.R.P., 530,649, (*Chem. Zentr.*, 1931, II, 1920).

Prins, *Rec. trav. chim.*, 1926, **45**, 80.

Kokatnur, *J. Am. Chem. Soc.*, 1919, **41**, 123.

***sym.*-Tetrachloroethane** (1 : 1 : 2 : 2-*Tetrachloroethane, acetylene tetrachloride*)

$CHCl_2{\cdot}CHCl_2$

$C_2H_2Cl_4$ MW, 168

Colourless liq. with odour similar to $CHCl_3$. F.p. − 43·8°. M.p. − 36°. B.p. 146·2°, 62°/45 mm., 55°/17 mm. D_4^{25} 1·5881, D^{15} 1·60255. n_D^{15} 1·49678. Volatile in steam. Sp. heat 0·286 cal./gm. at 20°. Solvent for P, S, fats, resins, cellulose acetate, rubber, etc. Non-inflammable. Alkalis ⟶ trichloroethylene. H_2O + Fe or Al ⟶ *sym.*-dichloroethylene. $AlCl_3$ at 110° ⟶ 1 : 1 : 1 : 2-tetrachloroethane. Narcotic and poisonous.

Ruhrchemie A.G., U.S.P., 2,016,658, (*Chem. Abstracts*, 1935, **29**, 8005).

Favorskiĭ, Margules, Davuidova, *Chem. Abstracts*, 1935, **29**, 7271.

Frydlender, *Chem. Abstracts*, 1935, **29**, 7935.

I.G., F.P., 739,183, (*Chem. Zentr.*, 1933, I, 2312).

Timmermans, *Bull. soc. chim. Belg.*, 1927, **36**, 502.

Tetrachloroethylene (*Perchloroethylene*)

$CCl_2{:}CCl_2$

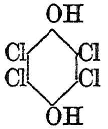
C_2Cl_4 MW, 166

Liq. F.p. — 22·35°. M.p. — 19°. B.p. 121·20°, 33·2°/30 mm. D^0 1·65582. n_D^{20} 1·50180. Heat of comb. C_p 162·5 Cal. Sp. heat at 20° 0·216 cal./gm. Cl ⟶ hexachloroethane. Vapour is poisonous. Extensively used as industrial solvent. Non-inflammable.

Frydlender, *Chem. Abstracts*, 1935, **29**, 7935.
Reilly, U.S.P., 1,947,491, (*Chem. Abstracts*, 1934, **28**, 2371).
Timmermans, Hennault-Roland, *J. chim. phys.*, 1930, **27**, 401.
Thusen, U.S.P., 1,590,265, (*Chem. Abstracts*, 1926, **20**, 3015).
Weisen, Wightman, *J. Phys. Chem.*, 1919, **23**, 415.

Tetrachloroguaiacol.
See under Tetrachlorocatechol.

Tetrachlorohydroquinone

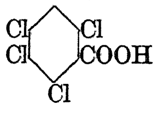


$C_6H_2O_2Cl_4$ MW, 248

Needles from AcOH. M.p. 232°. Sol. EtOH, Et_2O. Insol. H_2O, CCl_4, CS_2, C_6H_6. Sublimes.
Di-Me ether: $C_8H_6O_2Cl_4$. MW, 276. Needles. M.p. 164°. Sol. EtOH, AcOH. Mod. sol. Et_2O. Sublimes.
Me-Et ether: $C_9H_8O_2Cl_4$. MW, 290. Needles from EtOH. M.p. 101°. Sol. Et_2O, C_6H_6, ligroin. Mod. sol. EtOH, AcOH. Sublimes.
Di-Et ether: $C_{10}H_{10}O_2Cl_4$. MW, 304. Needles. M.p. 112°. Sol. EtOH, Et_2O. Sublimes.
Diacetyl: needles from AcOH. M.p. 251°.
Dipropionyl: cryst. M.p. 160°.
Dibutyryl: cryst. M.p. 137°.
Dibenzoyl: needles from CS_2. M.p. 232°.

Graebe, *Ann.*, 1868, **146**, 20.
Bouveault, *Compt. rend.*, 1899, **129**, 55.
Fiala, *Monatsh.*, 1885, **6**, 912.
Niemeyer, *Ann.*, 1885, **228**, 324.
Klinger, *Ann.*, 1911, **382**, 221.

Tetrachloroisophthalic Acid



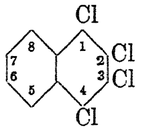
$C_8H_2O_4Cl_4$ MW, 304

Needles. M.p. 267–9°. Sol. EtOH. Very spar. sol. C_6H_6.

Rupp, *Ber.*, 1896, **29**, 1632.

Tetrachloromethane.
See Carbon tetrachloride.

1 : 2 : 3 : 4-Tetrachloronaphthalene



$C_{10}H_4Cl_4$ MW, 266

M.p. 198°.

v. Braun *et al.*, *Ber.*, 1923, **56**, 2337.
Wynne, *J. Chem. Soc.*, 1946, 61.

1 : 2 : 3 : 5-Tetrachloronaphthalene.
Needles from EtOH. M.p. 141°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 249.

1 : 2 : 3 : 7-Tetrachloronaphthalene.
Needles from EtOH. M.p. 115°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 250.

1 : 2 : 4 : 6-Tetrachloronaphthalene.
Needles from EtOH. M.p. 111°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 252.

1 : 2 : 5 : 6-Tetrachloronaphthalene.
Needles from EtOH. M.p. 164°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 247.
Piggott, Slinger, *J. Chem. Soc.*, 1952, 259.

1 : 3 : 5 : 7-Tetrachloronaphthalene.
Needles from EtOH. M.p. 179°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 253.

1 : 3 : 5 : 8-Tetrachloronaphthalene.
Needles from EtOH. M.p. 172°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 254.

1 : 3 : 6 : 7-Tetrachloronaphthalene.
Needles from EtOH. M.p. 120°.

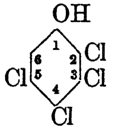
Turner, Wynne, *J. Chem. Soc.*, 1941, 253.

1 : 4 : 6 : 7-Tetrachloronaphthalene.
Prisms from ligroin. M.p. 136°.

Turner, Wynne, *J. Chem. Soc.*, 1941, 256.

Tetrachlorophenetole.
See under Tetrachlorophenol.

2 : 3 : 4 : 5-Tetrachlorophenol



$C_6H_2OCl_4$ MW, 232

Needles from ligroin. M.p. 116–17°.
Benzoyl: m.p. 110°.

Holleman, *Rec. trav. chim.*, 1921, **40**, 318.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

2 : 3 : 4 : 6-Tetrachlorophenol.
Needles from ligroin. M.p. 70°. B.p. 150°/15 mm. Sol. EtOH, $CHCl_3$, CS_2, C_6H_6, ligroin. Mod. sol. pet. ether. Spar. sol. H_2O.
Me ether: 2 : 3 : 4 : 6-tetrachloroanisole. $C_7H_4OCl_4$. MW, 246. Needles from MeOH. M.p. 64–5°.

Et ether: 2 : 3 : 4 : 6-tetrachlorophenetole. $C_8H_6OCl_4$. MW, 260. Needles from EtOH. M.p. 55°.

Acetyl: cryst. from EtOH. M.p. 65–6°.

Benzoyl: m.p. 108°.

Lock, *Monatsh.*, 1936, **67**, 320.
Holleman, *Rec. trav. chim.*, 1921, **40**, 318.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

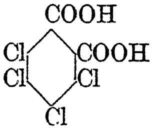
2 : 3 : 5 : 6-Tetrachlorophenol.

Leaflets from ligroin. M.p. 115°.

Benzoyl: m.p. 136°.

Holleman, *Rec. trav. chim.*, 1921, **40**, 318.
Tiessens, *Rev. trav. chim.*, 1931, **50**, 112.

Tetrachlorophthalic Acid



$C_8H_2O_4Cl_4$ MW, 304

Plates from H_2O. At 98° ⟶ anhydride. Very sol. Me_2CO. Sol. EtOH, Et_2O. Spar. sol. H_2O, $CHCl_3$, C_6H_6.

Mono-Me ester: $C_9H_4O_4Cl_4$. MW, 318. Needles. M.p. 142°.

Di-Me ester: $C_{10}H_6O_4Cl_4$. MW, 332. Prisms. M.p. 92°.

Mono-Et ester: $C_{10}H_6O_4Cl_4$. MW, 332. M.p. 94–5°.

Di-Et ester: $C_{12}H_{10}O_4Cl_4$. MW, 360. Cryst. M.p. 60·5°.

*Di-*n*-propyl ester*: b.p. 170–5°/1 mm. n_D^{25} 1·5348.

*Di-*n*-butyl ester*: b.p. 180–5°/1 mm. n_D^{25} 1·5292.

Dihexadecyl ester: plates from AcOEt. M.p. 49–50°.

Monobenzyl ester: m.p. 130–1°.

Dibenzyl ester: needles from MeOH. M.p. 92–3°.

*Di-*p*-nitrobenzyl ester*: needles from C_6H_6. M.p. 180–1°.

sym.-*Dichloride*: $C_8O_2Cl_6$. MW, 341. Prisms from pet. ether. M.p. 48°.

unsym.-*Dichloride*: needles from pet. ether. M.p. 137°.

Anhydride: $C_8O_3Cl_4$. MW, 286. Needles. M.p. 255°. Sublimes. Spar. sol. Et_2O.

Imide: $C_8HO_2NCl_4$. MW, 285. Plates from AcOH. M.p. 338–9°.

Anil: plates from AcOH. M.p. 268–9°.

o-*Nitroanil*: plates from C_6H_6. M.p. 272–3°.

m-*Nitroanil*: plates from AcOH–xylene. M.p. 300–301·5°.

p-*Nitroanil*: needles from C_6H_6. M.p. 292–7°.

o-*Tolil*: plates from AcOH. M.p. 232–236·5°.

m-*Tolil*: plates. M.p. 245·5–246·5°.

p-*Tolil*: exists in two forms. (i) Plates from AcOH. M.p. 207°. (ii) Yellow needles from AcOH. M.p. 214–15°.

β-*Naphthylimide*: m.p. 287°.

Salkind, Belikova, *Brit. Chem. Abstracts*, 1936, 331.
Kirpal, Kunze, *Ber.*, 1929, **62**, 2102.
Pratt, Perkins, *J. Am. Chem. Soc.*, 1918, **40**, 203.
Tingle, Bates, *J. Am. Chem. Soc.*, 1910, **32**, 1325.
Eckert, Steiner, *Monatsh.*, 1915, **36**, 272.
Graebe, *Ann.*, 1887, **238**, 332.
Meyer, Jugilewitsch, *Ber.*, 1897, **30**, 780.

1 : 1 : 1 : 2-Tetrachloropropane

$CH_3{\cdot}CHCl{\cdot}CCl_3$

$C_3H_4Cl_4$ MW, 182

Oil. M.p. – 65°. B.p. 152–3°. D^{20} 1·473. n_D^{20} 1·4867. Insol. H_2O.

Henry, *Rec. trav. chim.*, 1905, **24**, 333.
Kharasch, Rossin, Fields, *J. Am. Chem. Soc.*, 1941, **63**, 2560.

1 : 1 : 1 : 3-Tetrachloropropane

$CH_2Cl{\cdot}CH_2{\cdot}CCl_3$

$C_3H_4Cl_4$ MW, 182

Liq. B.p. 159°/760 mm., 59°/24 mm. D_4^{25} 1·4463. n_D^{25} 1·4794.

Joyce, Hanford, Harmon, *J. Am. Chem. Soc.*, 1948, **70**, 2529.

1 : 1 : 2 : 2-Tetrachloropropane

$CH_3{\cdot}CCl_2{\cdot}CHCl_2$

$C_3H_4Cl_4$ MW, 182

Liq. B.p. 153°. Misc. with EtOH, Et_2O. D^{13} 1·47. n_D^{25} 1·4850.

Borsche, Fittig, *Ann.*, 1865, **133**, 114.
Huntress, Sanchez-Nieva, *J. Am. Chem. Soc.*, 1948, **70**, 2814.

1 : 1 : 2 : 3-Tetrachloropropane

$CH_2Cl{\cdot}CHCl{\cdot}CHCl_2$

$C_3H_4Cl_4$ MW, 182

Liq. B.p. 179–80°/756·6 mm. D^{15} 1·521. n_D^{17} 1·5037.

Hartenstein, *J. prakt. Chem.*, 1873, **7**, 313.
Mouneyrat, *Bull. soc. chim.*, 1900, **21**, 621.
Kirrmann, Kremer, *Bull. soc. chim.*, 1948, 168.

1 : 2 : 2 : 3-Tetrachloropropane

$CH_2Cl{\cdot}CCl_2{\cdot}CH_2Cl$

$C_3H_4Cl_4$ MW, 182

Liq. B.p. 164°. D^{17} 1·496. n_D^{18} 1·4940.

Herzfelder, *Ber.*, 1893, **26**, 2436.
Kirrmann, Kremer, *Bull. soc. chim.*, 1948, 167.

1 : 2 : 3 : 3-Tetrachloropropylene

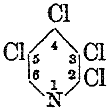
$CHCl_2{\cdot}CCl{:}CHCl$

$C_3H_2Cl_4$ MW, 180

B.p. 165°. D_4^{24} 1·5274. n_D^{18} 1·5272.

Prins, *J. prakt. Chem.*, 1914, **89**, 421.
Skoldinov, Oseled'ko, Russ. P. 66,825, (*Chem. Abstracts*, 1947, **41**, 4801).

2 : 3 : 4 : 5-Tetrachloropyridine



C_5HNCl_4 MW, 217

Needles from EtOH. M.p. 21–2°. B.p. 135–7°/24 mm. Volatile in steam.

Sell, Dootson, *J. Chem. Soc.*, 1899, **75**, 986; 1900, **77**, 3.

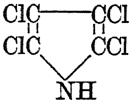
2 : 3 : 4 : 6-Tetrachloropyridine.
Plates from 50% EtOH. M.p. 74–5°. B.p. 130–5°/16–20 mm. Insol. H_2O, acids.

Sell, Dootson, *J. Chem. Soc.*, 1898, **73**, 440; 1900, **77**, 1.

2 : 3 : 5 : 6-Tetrachloropyridine.
Cryst. from EtOH.Aq. M.p. 90–1°. B.p. 250–1°, 125–30°/16–20 mm. Sol. EtOH, Et_2O, pet. ether. Insol. H_2O, acids.

Sell, Dootson, *J. Chem. Soc.*, 1897, **71**, 1081; 1898, **73**, 439.

2 : 3 : 4 : 5-Tetrachloropyrrole



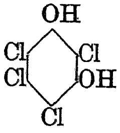
C_4HNCl_4 MW, 205

Plates from pet. ether. M.p. 110°. Sol. EtOH, Et_2O. Spar. sol. H_2O.
N-Me: $C_5H_3NCl_4$. MW, 219. Needles from pet. ether. M.p. 118–19°. Spar. sol. H_2O. Volatile in steam.
N-Phenyl: $C_{10}H_5NCl_4$. MW, 281. Cryst. from ligroin. M.p. 93°.

Anschütz, Beavis, *Ann.*, 1897, **295**, 30.
Mazzara, Borgo, *Gazz. chim. ital.*, 1904, **34**, i, 258.
Kalle, D.R.P., 38,423, (*Chem. Zentr.*, 1887, 423).
Ciamician, Silber, *Ber.*, 1885, **18**, 1763.

Tetrachloroquinone.
See Chloranil.

Tetrachlororesorcinol

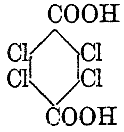


$C_6H_2O_2Cl_4$ MW, 248

Needles from H_2O. M.p. 141°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Mod. sol. H_2O.
Di-Et ether: $C_{10}H_{10}O_2Cl_4$. MW, 304. Needles from EtOH. M.p. 73°. Sol. Et_2O, $CHCl_3$, AcOH, C_6H_6, ligroin. Spar. sol. EtOH.
Dipropyl ether: $C_{12}H_{14}O_2Cl_4$. MW, 332. Oil. Decomp. at 100°. Sol. EtOH, Et_2O, AcOH.
Diacetyl: needles. M.p. 145°.

Zincke, Fuchs, *Ber.*, 1892, **25**, 2689.
Jackson, Carlton, *Am. Chem. J.*, 1914, **31**, 379.

Tetrachloroterephthalic Acid



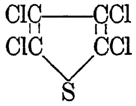
$C_8H_2O_4Cl_4$ MW, 304

Cryst. from H_2O. M.p. 343–5° decomp. Sol. EtOH, Et_2O, AcOH. Insol. H_2O, C_6H_6.
Di-Me ester: needles from MeOH. M.p. 154–5°.
Chloride: cryst. from CCl_4. M.p. 144–5°.
Amide: cryst. from AcOH. Does not melt below 400°.

Qvist, Holmberg, *Chem. Zentr.*, 1932, II, 2816.
Rupp, *Ber.*, 1896, **29**, 1628.
Rabjohn, *J. Am. Chem. Soc.*, 1948, **70**, 3518.

Tetrachlorotetramethylmethane.
See Pentaerythrityl tetrachloride.

Tetrachlorothiophene

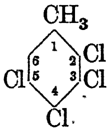


C_4Cl_4S MW, 222

Cryst. from EtOH.Aq. M.p. 29°. B.p. 240–5°, 75–7°/2 mm. D_4^{30} 1·7036. n_D^{30} 1·5915.

Weitz, *Ber.*, 1884, **17**, 795.
Rosenberg, *Ber.*, 1886, **19**, 650.
Coonradt, Hartough, *J. Am. Chem. Soc.*, 1948, **70**, 1160.

2 : 3 : 4 : 5-Tetrachlorotoluene



$C_7H_4Cl_4$ MW, 230

Needles from MeOH. M.p. 97–8°.

Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1285; 1906, **89**, 1454.

2 : 3 : 4 : 6-Tetrachlorotoluene.
Needles from EtOH. M.p. 91·5–92°.

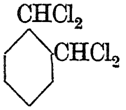
Bureš, Trpisovska, *Chem. Zentr.*, 1936, I, 1209.
Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1280.

2 : 3 : 5 : 6-Tetrachlorotoluene.
Needles from MeOH. M.p. 93–4°. Sublimes.

Qvist, Holmberg, *Chem. Zentr.*, 1932, II, 2816.
Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1281.

Tetrachloroveratrol.
See under Tetrachlorocatechol.

ω : ω : ω′ : ω′-Tetrachloro-*o*-xylene

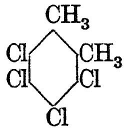


$C_8H_6Cl_4$ MW, 244.

Cryst. from Et_2O. M.p. 89°. B.p. 273–4°. D^0 1·601. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O. Boil with $H_2O \longrightarrow$ terephthaldehyde.

Hjelt, *Ber.*, 1885, **18**, 2879.
Colson, Gautier, *Ann. chim. phys.*, 1887, **11**, 25.
Arditte, *Compt. rend.*, 1946, **223**, 635.

3 : 4 : 5 : 6-Tetrachloro-*o*-xylene

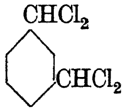


$C_8H_6Cl_4$ MW, 244

Needles from Et_2O. M.p. 215°. Sublimes. Sol. EtOH, Et_2O, C_6H_6. Non-volatile in steam.

Claus, Kautz, *Ber.*, 1885, **18**, 1369.
Datta, Chatterjee, *J. Am. Chem. Soc.*, 1917, **38**, 2549.

ω : ω : ω′ : ω′-Tetrachloro-*m*-xylene

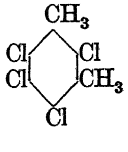


$C_8H_6Cl_4$ MW, 244

B.p. 273°.

Colson, Gautier, *Bull. soc. chim.*, 1886, **45**, 509.

2 : 4 : 5 : 6-Tetrachloro-*m*-xylene

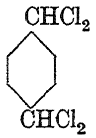


$C_8H_6Cl_4$ MW, 244

Needles from EtOH–$CHCl_3$. M.p. 219° (212°). Sol. Et_2O, $CHCl_3$, C_6H_6. Insol. EtOH.

Datta, Fernandes, *J. Am. Chem. Soc.*, 1917, **38**, 1810.
Bureš, Borgmann, *Chem. Zentr.*, 1928, I, 1171.

ω : ω : ω′ : ω′-Tetrachloro-*p*-xylene

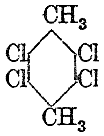


$C_8H_6Cl_4$ MW, 244

Cryst. from Et_2O. M.p. 93°. D^0 1·606.

Colson, Gautier, *Ann. chim. phys.*, 1887, **11**, 24.

2 : 3 : 5 : 6-Tetrachloro-*p*-xylene



$C_8H_6Cl_4$ MW, 244

Needles from AcOH. M.p. 216·5–217°. Sol. EtOH, Et_2O, C_6H_6.

Bureš, Rubeš, *Chem. Zentr.*, 1929, I, 507.
Datta, Fernandes, *J. Am. Chem. Soc.*, 1916, **38**, 1811.
Ruggli, Brandt, *Helv. Chim. Acta*, 1944, **27**, 282.

Tetracosandioic Acid.
See Docosane-1 : 22-dicarboxylic Acid.

Tetracosane

$$CH_3{\cdot}[CH_2]_{22}{\cdot}CH_3$$

$C_{24}H_{50}$ MW, 338

Isolated from paraffin from low-temperature coal tar. Cryst. from Et_2O. M.p. 54°. B.p. 237–40°/15 mm. (243–4°/15 mm.). n_D^{65} 1·43026, n_D^{80} 1·42448.

Levene, West, *J. Biol Chem.*, 1914, **18**, 478.
Gluud, *Ber.*, 1919, **52**, 1040.
Krafft, *Ber.*, 1882, **15**, 1718.

Tetracosane-1 : 24-dicarboxylic Acid (n-*Hexacosandioic acid*)

$$HOOC{\cdot}[CH_2]_{24}{\cdot}COOH$$

$C_{26}H_{50}O_4$ MW, 426

M.p. 123·5°.
Di-Et ester: m.p. 66°.

Fairweather, *Proc. Roy. Soc.*, 1926, **46**, 71.

Tetracosanic Acid (n-*Tetracosanoic acid*. See also Lignoceric Acid)

$$CH_3{\cdot}[CH_2]_{22}{\cdot}COOH$$

$C_{24}H_{48}O_2$ MW, 368.

Plates from AcOH. M.p. 87·5–88° (85–6°).
Anhydride: $C_{48}H_{94}O_3$. MW, 718. M.p. 86·0–86·3°.
Me ester: $C_{25}H_{50}O_2$. MW, 382. M.p. 59·5–60°.
Et ester: $C_{26}H_{52}O_2$. MW, 396. M.p. 56–7°. B.p. 198–9°/0·24 mm.

Nitrile: $C_{24}H_{47}N$. MW, 349. M.p. 61·2° (55–6°).

Meyer, Brod, Soyka, *Monatsh.*, 1913, **34**, 1133.
Levene, West, Allen, van der Scheer, *J. Biol. Chem.*, 1915, **23**, 75.
Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.
Francis, Piper, Malkin, *Proc. Roy. Soc.*, 1930, **128** A, 214.
Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2504.
Brigl, *Z. physiol. chem.*, 1915, **95**, 161.

Tetracosanol (1-*Hydroxytetracosane, tetracosyl alcohol*. See also Lignoceryl Alcohol)

$CH_3{\cdot}[CH_2]_{22}{\cdot}CH_2OH$

$C_{24}H_{50}O$ MW, 354.

M.p. 76·5–77·5°. B.p. 210–210·5°/0·4 mm.
Acetyl: m.p. 57°.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 915.
Taylor, Levene, *J. Biol. Chem.*, 1928, **80**, 609.
Brigl, Fuchs, *Z. physiol. chem.*, 1922, **119**, 280.
Bleyberg, Ulrich, *Ber.*, 1931, **64**. 2504.

14-Tetracosenic Acid

$CH_3{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_{13}{\cdot}COOH$

$C_{24}H_{46}O_2$ MW, 366

Cis:
See Nervonic Acid.
Trans:
Cryst. from EtOH. M.p. 61°.

Hale, Lycan, Adams, *J. Am. Chem. Soc.*, 1930, **52**, 4538.

Tetracosyl Alcohol.
See Tetracosanol.

Tetracosyl iodide (1-*Iodotetracosane*)

$CH_3{\cdot}[CH_2]_{22}{\cdot}CH_2I$

$C_{24}H_{49}I$ MW, 464

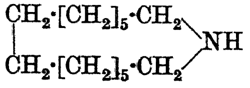
M.p. 54·5–55·5°. B.p. 207–9°/0·35 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 917.
Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2504.

Tetradecahydroacridine.
See Perhydroacridine.

Tetradecahydroanthracene.
See Perhydroanthracene.

Tetradecamethyleneimine



$C_{14}H_{29}N$ MW, 211

Cryst. M.p. 55°. pK 9·35.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1949, **32**, 544.

Tetradecane

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_3$

$C_{14}H_{30}$ MW, 198

M.p. 5·5°. B.p. 252·5°, 158°/50 mm., 129·5°/15 mm., 98·5°/1 mm. D_4^{20} 0·7645.

Maman, *Chem. Zentr.*, 1936, I, 2332.
Krafft, *Ber.*, 1882, **15**, 1700.
Sorabji, *J. Chem. Soc.*, 1885, **47**, 41.

Tetradecane-1 : 14-dicarboxylic Acid.
See Thapsic Acid.

Tetradecane-1-sulphonic Acid

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2{\cdot}SO_3H$

$C_{14}H_{30}O_3S$ MW, 278

M.p. 55–6°.
Monohydrate: m.p. 66·5°.
Benzylaniline salt: m.p. 82·5–82·7°.

Noller, Gordon, *J. Am. Chem. Soc.*, 1933, **55**, 1090.

Tetradecanol-1.
See Tetradecyl Alcohol.

Tetradecanol-3.
See Ethylundecylcarbinol.

Tetradecanone-2.
See Methyl dodecyl Ketone.

Tetradecanone-3.
See Ethyl undecyl Ketone.

Tetradecylacetylene (*Hexadecyne*-1)

$CH_3{\cdot}[CH_2]_{13}{\cdot}C{:}CH$

$C_{16}H_{30}$ MW, 222

M.p. 15°. B.p. 155°/15 mm. D^{20} 0·7965.

Krafft, *Ber.*, 1896, **29**, 2236.

Tetradecyl Alcohol (*Tetradecanol*-1, *myristyl alcohol*, 1-*hydroxytetradecane*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2OH$

$C_{14}H_{30}O$ MW, 214

Cryst. from EtOH.Aq. M.p. 39–39·5°. B.p. 170–3°/20 mm., 160°/10 mm. D_4^{38} 0·8236, D_4^{50} 0·8153.
Formyl: b.p. 166°/17 mm.
Acetyl: m.p. 14°. D_4^{25} 0·8581. n_D^{20} 1·4373.
Myristyl: plates from EtOH. M.p. 43°.
p-*Nitrobenzoyl*: m.p. 45°.
3 : 4 : 5-*Tri-iodobenzoyl*: m.p. 80–1°.
p-*Toluenesulphonyl*: m.p. 35°.
p-*Bromobenzenesulphonyl*: m.p. 51·5°.
p-*Methoxyphenylurethane*: m.p. 83°.
3 : 4-*Dimethoxyphenylurethane*: m.p. 79·5°.
Nitrate: liq. B.p. 175–80°/12 mm. slight decomp. Spar. sol. EtOH.

Ford, Marvel, *Organic Syntheses*, 1930, X, 64.
Adkins, Folkers, *J. Am. Chem. Soc.*, 1931, **53**, 1096.
Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, **55**, 3825.

Tetradecylaldehyde.
See Myristic Aldehyde.

Tetradecylamine (1-*Aminotetradecane*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2NH_2$

$C_{14}H_{31}N$ MW, 213

M.p. 37°. B.p. 162°/15 mm.
Acid succinate: needles. M.p. 123°.

Shukoff, Schestakoff, *J. prakt. Chem.*, 1903, **67**, 419.
Krafft, *Ber.*, 1890, **23**, 2361.

1-Tetradecylene

$CH_3{\cdot}[CH_2]_{11}{\cdot}CH{:}CH_2$

$C_{14}H_{28}$ MW, 196

M.p. − 12°. B.p. 232–4°/760 mm., 124·5–125°/15 mm. D_4^{15} 0·7745, D_4^{30} 0·7683. n_D^{22} 1·4392.

Klepper, *Chem. Abstracts*, 1929, **23**, 2897.
Krafft, *Ber.*, 1883, **16**, 3021.

1-Tetradecylenic Acid (1-*Tetradecenoic acid*, 1-*tridecylene*-1-*carboxylic acid*)

$CH_3{\cdot}[CH_2]_{10}{\cdot}CH{:}CH{\cdot}COOH$

$C_{14}H_{26}O_2$ MW, 226

M.p. 33° (50–3°). B.p. 186–8°/8 mm.
Et ester: b.p. 165–7°/11 mm.
Dibromide: m.p. 57–8°.

Kyrides, Zienty, U.S.P. 2,399,601; *Chem. Abstracts*, 1946, **40**, 4180; 1947, **41**, 7396.
Tulus, *Chem. Abstracts*, 1946, **40**, 3722.

3-Tetradecylenic Acid (3-*Tetradecenoic acid*, 3-*tridecylene*-1-*carboxylic acid*, *tsuzuic acid*, *tsuduic acid*)

$CH_3{\cdot}[CH_2]_8{\cdot}CH{:}CH{\cdot}[CH_2]_2{\cdot}COOH$

$C_{14}H_{26}O_2$ MW, 226

Occurs in tohaku oil (seed oil of *Lindera obtusiloba*) and in seed oil of "tsuzu" (*Tetradenia glauca*). M.p. 18–18·5°. B.p. 185–8°/13 mm. D_4^{20} 0·9024. n_D^{20} 1·4559.
Me ester: $C_{15}H_{28}O_2$. MW, 240. B.p. 158–60°/15 mm. D^{15} 0·8857. n_D^{15} 1·4489.
Et ester: $C_{16}H_{30}O_2$. MW, 254. B.p. 168–70°/15 mm. D^{15} 0·8783. n_D^{15} 1·4479.
p-*Bromophenacyl ester*: m.p. 61·3°.
p-*Phenylphenacyl ester*: m.p. 54·5°.

Toyama, *J. Soc. Chem. Ind. Japan*, 1937, **40**, 285.
Komori, Ueno, *Bull. Chem. Soc. Japan*, 1937, **12**, 433.
Tsujimoto, *Chem. Abstracts*, 1928, **22**, 4470.

4-Tetradecylenic Acid (*Physeteric acid*, 4-*tetradecenoic acid*, 4-*tridecylene*-1-*carboxylic acid*)

$CH_3{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_3{\cdot}COOH$

$C_{14}H_{26}O_2$ MW, 226

Occurs in sperm whale oil. M.p. 20°. D_4^{20} 0·9046. n_D^{20} 1·4552.

Tsujimoto, *J. Soc. Chem. Ind. Japan*, 1926, **29**, 102; *Chem. Abstracts*, 1929, **23**, 1109.
Toyama, Tsuchiya, *Chem. Abstracts*, 1936, **30**, 315.

8-Tetradecylenic Acid (8-*Tetradecenoic acid*, *myristoleic acid*, 8-*tridecylene*-1-*carboxylic acid*)

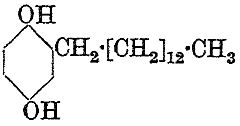
$CH_3{\cdot}[CH]_3{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}COOH$

$C_{14}H_{26}O_2$ MW, 226

Occurs in natural fats.
Me ester: b.p. 125–30°/3–5 mm.

Armstrong, Hilditch, *J. Soc. Chem. Ind.*, 1925, **44**, 180T.

Tetradecylhydroquinone (*Myristylquinol*, 2 : 5-*dihydroxytetradecylbenzene*)



$C_{20}H_{34}O_2$ MW, 306

Cryst. from pet. ether. M.p. 110°. Ag_2O ⟶ tetradecylbenzoquinone, m.p. 77·5°.
Mono-Me ether: needles from AcOH. M.p. 57–60°. B.p. 195°/0·15 mm.
Di-Me ether: 2 : 5-dimethoxytetradecylbenzene. B.p. 165°/0·5 mm.
Di-Et ether: 2 : 5-diethoxytetradecylbenzene. B.p. 183°/0·1 mm.

Cook, Heilbron, Lewis, *J. Chem. Soc.*, 1942, 660.
Asano, Hase, *Chem. Abstracts*, 1942, **36**, 82.

Tetradecylic Acid.
See Myristic Acid.

Tetradecyl iodide (1-*Iodotetradecane*)

$CH_3{\cdot}[CH_2]_{12}{\cdot}CH_2I$

$C_{14}H_{29}I$ MW, 324

B.p. 192–5°/17·5 mm., 128°/0·5 mm.

Levene, West, van der Scheer, *J. Biol. Chem.*, 1915, **20**, 529.
Majima, Nakamura, *Ber.*, 1913, **46**, 4094.

Tetradecylmalonic Acid (*Pentadecane*-1 : 1-*dicarboxylic acid*)

$CH_3{\cdot}[CH_2]_{13}{\cdot}CH{<}^{COOH}_{COOH}$

$C_{17}H_{32}O_4$ MW, 300

Cryst. from C_6H_6. M.p. 123–4° (117–18°). Very sol. boiling AcOH. Spar. sol. cold EtOH. Insol. Et_2O.
Di-Et ester: $C_{21}H_{40}O_4$. MW, 356. Oil. B.p. 190°/3 mm.
Monoamide: $C_{17}H_{33}O_3N$. MW, 299. Glistening cryst. Sol. boiling EtOH. Insol. H_2O, Et_2O. Heat ⟶ palmitic amide.
Mononitrile: 1-cyanopalmitic acid. $C_{17}H_{31}O_2N$. MW, 281. Leaflets from AcOH. M.p. 75–6°.

Chargaff, *Ber.*, 1932, **65**, 752.
Hell, Jordanow, *Ber.*, 1891, **24**, 990.

Tetraethylammonium bromide

$(C_2H_5)_4NBr$

$C_8H_{20}NBr$ MW, 210

Cryst. from EtOH. Very sol. H_2O, EtOH, $CHCl_3$. D_4^{25} 1·3880.

B,Br₂ : orange-red needles from EtOH. M.p. 78° ⟶ dark red liq.

Add. comp. with thiourea : $C_8H_{20}NBr + 2CH_4N_2S$. Prisms. M.p. 159–60°.

Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, **43**, 190.
Walden, *Chem. Zentr.*, 1912, I, 1958.

Tetraethylammonium chloride

$(C_2H_5)_4NCl$

$C_8H_{20}NCl$ MW, 165·5

Very hygroscopic. Very sol. H_2O, EtOH, $CHCl_3$, Me_2CO. D_4^{25} 1·1115.

B,4H₂O : prisms. M.p. 37·5°

Walden, *Chem. Zentr.*, 1912, I, 1958.
Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, **43**, 189.

Tetraethylammonium hydroxide

$(C_2H_5)_4N{\cdot}OH$

$C_8H_{21}ON$ MW, 147

Known only in solution and in form of hydrates. Aq. sol. reacts alkaline and has bitter taste. Sucrose + $CuSO_4$ ⟶ blue sol. Hydrolyses fats.

B,4H₂O : needles. M.p. 49–50°. Very sol. H_2O.

B,6H₂O : m.p. 55°.

Hofmann, *Ann.*, 1851, **78**, 263.
Crichton, *J. Chem. Soc.*, 1907, **91**, 1794.

Tetraethylammonium iodide

$(C_2H_5)_4NI$

$C_8H_{20}NI$ MW, 257

Cryst. from warm H_2O. Does not melt below 200°. Sol. H_2O. Mod. sol. $CHCl_3$. Insol. Et_2O. D^4 1·559.

B,I₂ : prisms. M.p. 143°. Sol. Me_2CO, hot EtOH. Insol. Et_2O.

B,I₆ : cryst. M.p. 108°.

Add. comp. with thiourea : $C_8H_{20}NI + 2CH_4N_2S$. M.p. 135°.

Walden, *Chem. Zentr.*, 1914, I, 603.
Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, **43**, 191.

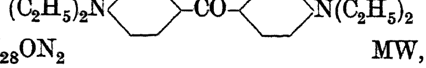
4 : 4′-Tetraethyldiaminobenzhydrol

$C_{21}H_{30}ON_2$ MW, 326

Cryst. from pet. ether. M.p. 78°.

Votoček, Köhler, *Ber.*, 1913, **46**, 1761.

4 : 4′-Tetraethyldiaminobenzophenone



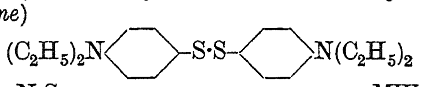
$C_{21}H_{28}ON_2$ MW, 324

Leaflets from EtOH. M.p. 95–6°. Intermediate for triphenylmethane dyes.

Anil : m.p. 124–5°.

Votoček, Köhler, *Ber.*, 1913, **46**, 1761.

4 : 4′-Tetraethyldiaminodiphenyl disulphide (*Dithiodiethylaniline*, *NN′-tetra-ethyldithioaniline*)

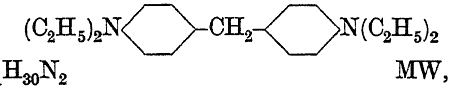


$C_{20}H_{28}N_2S_2$ MW, 360

Prisms from EtOH. M.p. 72°. Sol. CS_2. Spar. sol. EtOH, C_6H_6, ligroin, hot Et_2O.

Holzmann, *Ber.*, 1887, **20**, 1637.

4 : 4′ - Tetraethyldiaminodiphenylmethane



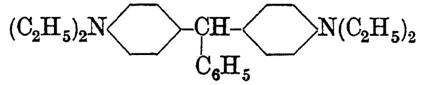
$C_{21}H_{30}N_2$ MW, 310

Cryst. from EtOH. M.p. 41°. B.p. 253°/10 mm.

Picrate : leaflets. M.p. 191°.

Votoček, Köhler, *Ber.*, 1913, **46**, 1760.
v. Braun, Kruber, *Ber.*, 1912, **45**, 2996.

4 : 4′ - Tetraethyldiaminotriphenylmethane



$C_{27}H_{34}N_2$ MW, 386

Needles from EtOH. M.p. 62°. Sol. EtOH, Et_2O, C_6H_6. Very spar. sol. H_2O.

Chloroplatinate : orange-red needles. M.p. 254–7°.

Doebner, *Ann.*, 1883, **217**, 265.
Decker, Becker, *Ann.*, 1913, **395**, 372.

***N* : *N′*-Tetraethyldithioaniline.**

See Tetraethyldiaminodiphenyl disulphide.

Tetraethylene Glycol (*Diethyl ether di-[β-hydroxyethyl] ether*)

$HO{\cdot}CH_2{\cdot}[CH_2{\cdot}O{\cdot}CH_2]_3{\cdot}CH_2OH$

$C_8H_{18}O_5$ MW, 194

B.p. 328°, 198°/14 mm., 157°/2 mm. D_4^{15} 1·1285. n_D^{15} 1·4609.

Cyclic carbonate : m.p. 42–4°. B.p. 128–30°/1 mm. D_4^{50} 1·1961. n_D^{50} 1·4569. Has slight odour of musk. Employed as perfume.

du Pont, F.P., 768,807, (*Chem. Abstracts*, 1935, **29**, 557).
Matignon, Moureu, Dodé, *Bull. soc. chim.*, 1934, **1**, 1308.
Hill, Carothers, *J. Am. Chem. Soc.*, 1933, **55**, 5034.

Tetraethylnaphthylenediamine.

See under Naphthylenediamine.

Tetraethylphenylenediamine.

See under Phenylenediamine.

Tetraethyl pyrophosphate (T.E.P.P.)

$[(C_2H_5O)_2PO{\cdot}O]_3PO$

$C_{12}H_{30}O_{13}P_4$ MW, 506

Oil. B.p. 145–50°/5–10 mm. Insecticide.

Sawhney, Seshadri, *Journal of Scientific and Industrial Research* (*India*), 1952, **11***B*, 348.

Tetraethylsilane.
See Silicon tetraethyl.

Tetraethylsuccinic Acid

$(C_2H_5)_2C{\cdot}COOH$
$(C_2H_5)_2C{\cdot}COOH$

$C_{12}H_{22}O_4$ MW, 230

Cryst. from Et_2O–pet. ether. M.p. 149° ⟶ anhydride. Very sol. Et_2O. Sol. EtOH. Spar. sol. H_2O, pet. ether. $k = 4{\cdot}4 \times 10^{-4}$ at 25°.

Di-Et ester: $C_{16}H_{30}O_4$. MW, 286. B.p. 168–72°/25 mm. $D_4^{?}$ 1·011.

Diamide: $C_{12}H_{24}O_2N_2$. MW, 228. Cryst. from pet. ether. M.p. 49–50°. B.p. 276–7°.

Dinitrile: $C_{12}H_{20}N_2$. MW, 192. Plates. M.p. 100–1°.

Anhydride: $C_{12}H_{20}O_3$. MW, 212. Prisms from pet. ether, with odour resembling camphor. M.p. 86°. B.p. 270°.

Verkade, Hartmann, *Rec. trav. chim.*, 1933, **52**, 945.
Walker, Walker, *J. Chem. Soc.*, 1905, **87**, 964.

Tetraethylthiourea.
See under Thiourea.

Tetraethylthiuram disulphide (*Antabuse*)

$(C_2H_5)_2N{\cdot}CS{\cdot}S{\cdot}S{\cdot}SC{\cdot}N(C_2H_5)_2$

$C_{10}H_{20}N_2S_4$ MW, 296

Cryst. M.p. 70°. Sol. warm EtOH. Spar. sol. Et_2O. Insol. H_2O. KOH fusion ⟶ diethylamine. Rubber vulcanisation accelerator. Antialcoholic drug.

Cummings, Simmons, *Ind. Eng. Chem.*, 1928, **20**, 1173.
Naugatuck, U.S.P., 1,782,111, (*Chem. Abstracts*, 1931, **25**, 303).
Roessler and Hasslacher, U.S.P., 1,796,977, (*Chem. Abstracts*, 1931, **25**, 2598).
Blake, *J. Am. Chem. Soc.*, 1943, **65**, 1268.
Hald *et al.*, *Chem. Abstracts*, 1949, **43**, 4379.
Hald, Jacobsen, *Lancet*, 1948, **225**, 1001.

Tetraethylurea.
See under Urea.

Tetrafluoroethylene

$F_2C{:}CF_2$

C_2F_4 MW, 100

B.p. – 78·4°.

Brode, Locke, Henne, *J. Am. Chem. Soc.*, 1934, **56**, 1726.
Booth *et al.*, *J. Am. Chem. Soc.*, 1933, **55**, 2231.
Park, *Ind. Eng. Chem.*, 1947, **31**, 354.

Tetrafluoromethane.
See Carbon tetrafluoride.

Tetrafluorosuccinic Acid (*Perfluorosuccinic acid*)

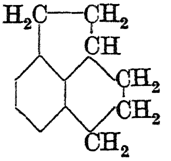
$HOOC{\cdot}CF_2{\cdot}CF_2{\cdot}COOH$

$C_4H_2O_4F_4$ MW, 190

Cryst. M.p. 86·4–87·4°. B.p. 150°/15 mm.

Henne, Zimmerschied, *J. Am. Chem. Soc.*, 1947, **69**, 281.

Tetrahydroacenaphthene (*Tetraphthene*)



$C_{12}H_{14}$ MW, 158

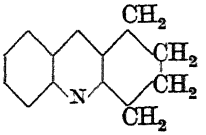
M.p. 12°. B.p. 249·5°/719 mm., 115°/12 mm. Turns slightly yellow in air. D^{21} 1·0290. n_D 1·5777. Decolourises $KMnO_4$. Reacts very vigorously with $CrO_3 + H_2SO_4$.

Picrate: m.p. about 152–3°.

Padoa, Fabris, *Gazz. chim. ital.*, 1909, **39**, i, 331.
Bayer, D.R.P., 306,724, (*Chem. Zentr.*, 1918, II, 420).
v. Braun, Kirschbaum, *Ber.*, 1922, **55**, 1682.
Goswami, *Compt. rend.*, 1924, **179**, 1269.
Fleischer, Siefert, *Ann.*, 1921, **422**, 304.

Δ¹-Tetrahydroacetophenone.
See 1-Acetocyclohexene.

1 : 2 : 3 : 4-Tetrahydroacridine



$C_{13}H_{13}N$ MW, 183

Cryst. from pet. ether. M.p. 54·5°.

B,HNO_3: pale yellow plates from EtOH. M.p. 160°.

B_2,H_2SO_4: needles from EtOH. M.p. 220°.

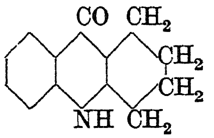
B_2,H_2PtCl_6: brownish-yellow needles from alc. HCl. M.p. 233–5°.

Picrate: yellow needles. M.p. 208° decomp.

Methiodide: needles from EtOH–Et_2O. M.p. 202–4°.

Perkin, Sedgwick, *J. Chem. Soc.*, 1924, **125**, 2446.
Borsche, *Ber.*, 1908, **41**, 2206.

1 : 2 : 3 : 4-Tetrahydroacridone



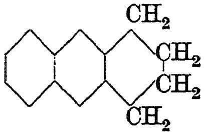
$C_{13}H_{13}ON$ MW, 199

Needles from EtOH or aniline. M.p. 358°. B.p. 360–70°/10 mm. Very sol. boiling aniline. Sol. EtOH with green fluor. Sublimation in air, or heat with H_2SO_4 ⟶ acridone.

Tiedtke, *Ber.*, 1909, **42**, 624.
Perkin, Sedgwick, *J. Chem. Soc.*, 1924, **125**, 2441.
v. Braun, Heymons, Manz, *Ber.*, 1931, **64**, 233.
Riedel, de Haen A.G., D.R.P., 532,397, (*Chem. Abstracts*, 1932, **26**, 151).

Tetrahydroanisole.
See under Cyclohexenol.

1 : 2 : 3 : 4-Tetrahydroanthracene (*Tethracene*)



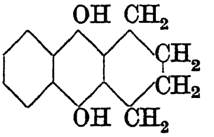
$C_{14}H_{14}$ MW, 182

Leaflets from EtOH. M.p. 103–5°. B.p. 170–3°/14 mm. CrO_3 in AcOH ⟶ 1 : 2 : 3 : 4-tetrahydroanthraquinone.

Picrate : reddish-yellow needles from EtOH. M.p. 116–17°.

Schroeter, *Ber.*, 1924, **57**, 2013; D.R.P., 463,830, (*Chem. Abstracts*, 1928, **22**, 4134).
Klepper, *Chem. Abstracts*, 1929, **23**, 3897.
Prokopetz, Khadzhinov, *Chem. Abstracts*, 1935, **29**, 7319.

1 : 2 : 3 : 4-Tetrahydroanthrahydroquinone (9 : 10-*Dihydroxy*-1 : 2 : 3 : 4-*tetrahydroanthracene*)



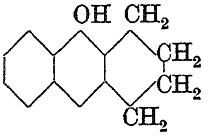
$C_{14}H_{14}O_2$ MW, 214

Greenish needles from AcOH. M.p. 208–16° decomp. Sol. alkalis with yellow col. Conc. H_2SO_4 ⟶ reddish-brown sol.

Diacetyl : cryst. from AcOH or EtOH. M.p. 204–6°.

Skita, *Ber.*, 1925, **58**, 2694.

1 : 2 : 3 : 4-Tetrahydroanthranol (9-*Hydroxy*-1 : 2 : 3 : 4-*tetrahydroanthracene*)



$C_{14}H_{14}O$ MW, 198

Pale yellow cryst. from ligroin. M.p. 108°. Sol. EtOH, Et_2O, AcOH. Very spar. sol. pet. ether. Sol. alkalis with strong yellowish-green fluor. No col. with $FeCl_3$.

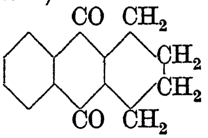
Me ether : $C_{15}H_{16}O$. MW, 212. Reddish-yellow oil. B.p. 197°/14 mm., slight decomp.

Acetyl : needles from EtOH. M.p. 109°. Spar. sol. EtOH with violet fluor.

Benzoyl : m.p. 142°.

v. Braun, Bayer, *Ber.*, 1925, **58**, 2675.

1 : 2 : 3 : 4 - Tetrahydroanthraquinone (*Tethracenequinone*)

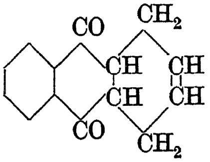


$C_{14}H_{12}O_2$ MW, 212

Golden-yellow needles from AcOH or AcOEt. M.p. 156–8°.

Schroeter, *Ber.*, 1924, **57**, 2014; D.R.P., 463,830, (*Chem. Abstracts*, 1928, **22**, 4134).
Skita, *Ber.*, 1925, **58**, 2694.

Δ2-Tetrahydroanthraquinone

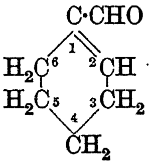


$C_{14}H_{12}O_2$ MW, 212

Cryst. from EtOH or ligroin. M.p. 102–3°. Sol. usual solvents on warming.

Diels, Alder, *Ann.*, 1928, **460**, 110.

Δ1-Tetrahydrobenzaldehyde (*Cyclohexene-1-aldehyde*)



$C_7H_{10}O$ MW, 110

Oil.

Oxime : cryst. from ligroin. M.p. 97–9°.
Semicarbazone : cryst. M.p. 212–13°.

Borsche, Schmidt, *Ber.*, 1910, **43**, 3400.
v. Braun, Danziger, *Ber.*, 1913, **46**, 107.
Wallach, *Ann.*, 1908, **359**, 292.

Δ³-Tetrahydrobenzaldehyde (*Cyclohexene-4-aldehyde*).

Oil. F.p. − 96·1°. B.p. 163·5–164·5°, 58°/17 mm., 51–2°/13 mm. D_4^{15} 0·9524. n_D^{19} 1·4725. Polymerises very readily.

Oxime: (i) Oil. B.p. 128–9°/25 mm. (ii) Cryst. M.p. 75–6°.

Semicarbazone: cryst. from C_6H_6–ligroin or MeOH.Aq. M.p. 153·5–154·5°.

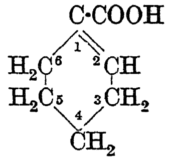
Phenylhydrazone: b.p. 207–8°/22 mm.

p-*Nitrophenylhydrazone*: m.p. 163°.

Sobecki, *Ber.*, 1910, **43**, 1040.
Diels, Alder, *Ann.*, 1928, **460**, 121.

Tetrahydrobenzene.
See Cyclohexene.

Δ¹-Tetrahydrobenzoic Acid (*Cyclohexene-1-carboxylic acid*)



$C_7H_{10}O_2$ MW, 126

Plates. M.p. 38°. B.p. 238–40°/683 mm., 138°/14 mm., 133–4°/11 mm., 107°/3 mm. $D_4^{47·2}$ 1·0717. n_{He}^{20} 1·49023.

Me ester: $C_8H_{12}O_2$. MW, 140. B.p. 193·5–194·5°. D_4^4 1·05607, D_{20}^{20} 1·04364.

Et ester: $C_9H_{14}O_2$. MW, 154. B.p. 206–8°, 143°/100 mm., 96°/16 mm., 84–6°/12 mm. D_4^{14} 1·0032. n_{He}^{20} 1·47167.

l-*Menthyl ester*: b.p. 178°/12 mm. $[\alpha]_D^{20}$ − 74·64° in EtOH.

Chloride: C_7H_9OCl. MW, 144·5. B.p. 86°/11 mm.

Nitrile: C_7H_9N. MW, 107. B.p. 81°/12 mm.

Amide: $C_7H_{11}ON$. MW, 125. Prisms from EtOH.Aq. M.p. 127–8°.

Darzens, Rost, *Compt. rend.*, 1911, **153**, 773.
Auwers, Krollpfeiffer, *Ber.*, 1915, **48**, 1396.
Ruzicka, Brugger, *Helv. Chim. Acta*, 1926, **9**, 402.
Boorman, Linstead, *J. Chem. Soc.*, 1935, 261.

Δ²-Tetrahydrobenzoic Acid (*Cyclohexene-3-carboxylic acid*).

Liq. B.p. 234–5°, 130°/18 mm. D_4^{20} 1·0820. n_D^{20} 1·4814. 1·34 parts sol. 100 parts H_2O at 20°. $k = 3·05 \times 10^{-5}$ at 25°. Oxidises slowly in air to benzoic acid.

Me ester: b.p. 188–9°. D_{20}^{20} 1·0433.

l-*Menthyl ester*: b.p. 176°/12 mm. $[\alpha]_D^{20}$ − 59·44° in EtOH.

Amide: leaflets or prisms from EtOH. M.p. 144°.

Boorman, Linstead, *J. Chem. Soc.*, 1935, 261.
Aschan, *Ann.*, 1892, **271**, 234.

Δ³-Tetrahydrobenzoic Acid (*Cyclohexene-4-carboxylic acid*).

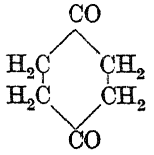
M.p. 17°. B.p. 237°/748 mm., 132·5–133°/20 mm. D_4^{20} 1·0815. n_D^{20} 1·4812. Oxidises in air very slowly.

Et ester: b.p. 194–5°. D_4^{20} 0·9688. n_D^{20} 1·4578.

Dibromide: m.p. 86°.

Perkin, Tattersall, *J. Chem. Soc.*, 1907, **91**, 490.
Sobecki, *Ber.*, 1910, **43**, 1039.

Tetrahydro-*p*-benzoquinone (*Cyclohexandione*-1 : 4, 1 : 4-*diketocyclohexane*)



$C_6H_8O_2$ MW, 112

Needles from pet. ether, plates from H_2O. M.p. 78°. Sol. usual solvents. Warm $FeCl_3$ ⟶ *p*-benzoquinone.

Di-Me acetal: m.p. 80–1°.

Di-Et acetal: plates from EtOH. M.p. 89°.

Dioxime: cryst. from H_2O. M.p. 188°. Mod. sol. H_2O.

Monosemicarbazone: cryst. from EtOH. M.p. 221–2° decomp. Mod. sol. EtOH.

Di-semicarbazone: m.p. 231°.

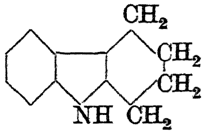
Di-thiosemicarbazone: cryst. M.p. 210–15°.

Di-2 : 4-dinitrophenylhydrazone: deep yellow cryst. from $PhNO_2$ or Py. M.p. 240°. Spar. sol. in EtOH, AcOEt.

Meerwein, *Ann.*, 1913, **398**, 248.
Baeyer, *Ann.*, 1894, **278**, 91.
Stollé, *Ber.*, 1901, **34**, 1344.
Piloty, Steinbock, *Ber.*, 1902, **35**, 3109.

Tetrahydroberberine.
See Canadine.

Tetrahydrocarbazole



$C_{12}H_{13}N$ MW, 171

Leaflets from EtOH.Aq. M.p. 120° (116°). B.p. 325–30°. Turns brown in air. Very sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Volatile in superheated steam. KOH fusion ⟶ indole-2-carboxylic acid.

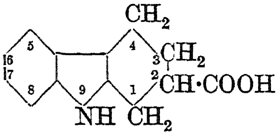
N-*Acetyl*: prisms from EtOH. M.p. 77°.

N-*Benzoyl*: m.p. 85°.

Picrate: dark red leaflets. M.p. 147°.

Bucherer, Brandt, *J. prakt. Chem.*, 1934, **140**, 129.
Perkin, Plant, *J. Chem. Soc.*, 1921, **119**, 1825.
Chem. Fabr. Weiler ter Meer, D.R.P., 374,098, (*Chem. Abstracts*, 1924, **18**, 2175).

Tetrahydrocarbazole-2-(or 4-)carboxylic Acid



$C_{13}H_{13}O_2N$ MW, 215

Cryst. M.p. 230°.

Baeyer, Tutein, *Ber.*, 1889, **22**, 2185.

Tetrahydrocarbazole-3-carboxylic Acid.

Cryst. from C_6H_6. M.p. 195°. Sol. EtOH. Spar. sol. $CHCl_3$, C_6H_6, pet. ether.

Perkin, *J. Chem. Soc.*, 1904, **85**, 428.

Tetrahydrocarbazole-5-(or 7-)carboxylic Acid.

Prisms from AcOH. M.p. 287°.

Me ester: needles from MeOH. M.p. 155°.

Et ester: plates from EtOH.Aq. M.p. 146°.

Perkin, *J. Chem. Soc.*, 1904, **85**, 428.

Tetrahydrocarbazole-6-carboxylic Acid.

Plates from EtOH.Aq. M.p. 282°.

Me ester: leaflets from AcOH.Aq. M.p. 158°.

Et ester: prisms from EtOH.Aq. M.p. 119°.

Perkin, *J. Chem. Soc.*, 1904, **85**, 428.

Tetrahydrocarbazole-7-(or 5-)carboxylic Acid.

Prisms from AcOH. M.p. 210°.

Me ester: prisms from EtOH.Aq. M.p. 93°.

Perkin, *J. Chem. Soc.*, 1904, **85**, 428.

Tetrahydrocarbazole-8-carboxylic Acid.

Prisms from C_6H_6. M.p. 203°.

Me ester: $C_{14}H_{15}O_2N$. MW, 229. Prisms from MeOH. M.p. 124°.

Et ester: $C_{15}H_{17}O_2N$. MW, 243. Needles from EtOH.Aq. M.p. 76°.

Collar, Plant, *J. Chem. Soc.*, 1926, 809.

Tetrahydrocarbazole-9-carboxylic Acid.

Not isolated as pure comp. owing to ease with which it loses CO_2.

Et ester: needles from EtOH. M.p. 65°.

Perkin, Plant, *J. Chem. Soc.*, 1923, **123**, 691.

Tetrahydrocarveol.

See Carvomenthol.

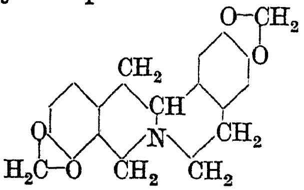
Tetrahydrocarvone.

See *p*-Menthanone-2.

Tetrahydrocinchoninic Acid.

See Tetrahydroquinoline-4-carboxylic Acid.

Tetrahydrocoptisine



$C_{19}H_{17}O_4N$ MW, 323

l-. Corydalis-D.

Occurs in Chinese *Corydalis ambigua*. Prisms. M.p. 204°. $[\alpha]_D^{25}$ − 295°.

B,HCl: m.p. about 250°.

B,HBr: m.p. about 260°.

dl-. Corydalis-E.

From Chinese *Corydalis ambigua*. Needles from $CHCl_3$. M.p. 222–3°. B.p. 260°/0·01 mm.

B,HCl: m.p. 246°.

Späth, Posega, *Ber.*, 1929, **62**, 1032.

Huang-Minlon, *Ber.*, 1936, **69**, 1737.

Chou, *Chinese Journal of Physiology*, 1936, **10**, 507, (*Chem. Abstracts*, 1937, **31**, 1161).

1 : 2 : 3 : 6-Tetrahydro-ψ-cumene.

See 1 : 4 : 5-Trimethylcyclohexene.

Tetrahydrocuminic Acid.

See Phellandric Acid.

Tetrahydrocymene.

See Menthene.

Tetrahydrodiphenyl.

See Phenylcyclohexene.

Tetrahydro-2 : 3-dipyridyl.

See Anatabine.

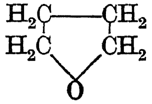
Tetrahydroeucarvone.

See 2 : 6 : 6-Trimethylcycloheptanone.

Tetrahydroferulene.

See under Ferulene.

Tetrahydrofuran (*Tetramethylene oxide*)



C_4H_8O MW, 72

Oil. F.p. − 65°. B.p. 64–5°. Sol. most org. solvents. Mod. sol. H_2O. D_4^{21} 0·888. n_D^{21} 1·40762. Bitter taste.

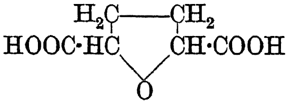
Starr, Hixon, *Organic Syntheses*, 1936, XVI, 77.

Bourguignon, *Chem. Zentr.*, 1908, I, 1630.

Tetrahydrofuran-carboxylic Acid.

See Tetrahydropyromucic Acid.

Tetrahydrofuran-2 : 5-dicarboxylic Acid



$C_6H_8O_5$ MW, 160

Cis:

Cryst. from AcOEt–pet. ether. M.p. 124–5°. Very sol. H_2O, EtOH, Me_2CO, AcOH. Mod. sol. Et_2O. Insol. $CHCl_3$, C_6H_6, pet. ether.

Di-Me ester: b.p. 90° (bath temp.)/0·03 mm. n_D^{15} 1·4550.

Diamide: cryst. from Me_2CO–MeOH. M.p. 189°.

Dianilide: plates from EtOH. M.p. 208–9° decomp. Sol. Me_2CO, $CHCl_3$. Spar. sol. EtOH, hot C_6H_6. Insol. H_2O.

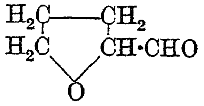
Anhydride: needles from $CHCl_3$–pet. ether. M.p. 128–9°. Sol. Et_2O, $CHCl_3$, AcOEt. Insol. pet. ether. Sublimes readily.

Trans:

Cryst. + H_2O from H_2O. M.p. 59–61°, anhyd. 94–5°. Very sol. H_2O, EtOH, Me_2CO, AcOH. Mod. sol. boiling Et_2O. Spar. sol. boiling C_6H_6, toluene, pet. ether.

Lean, *J. Chem. Soc.*, 1900, **77**, 110.
Le Sueur, Haas, *J. Chem. Soc.*, 1910, **97**, 181.

Tetrahydrofurfural (*Tetrahydro-2-furoic aldehyde*)



$C_5H_8O_2$ MW, 100

Mobile oil. B.p. 142–3°/779 mm., 45–7°/29 mm. D_4^{20} 1·0727. n_D^{20} 1·43658. Reduces hot Fehling's instantly, $NH_3.AgNO_3$ on standing. Decolourises alk. $KMnO_4$. Conc. HCl ⟶ intense brick-red col.

Di-Et acetal: b.p. 194·5° (187–90°).

Di-[β-ethoxyethyl]-acetal: b.p. 131–6°/4–5 mm.

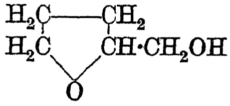
Diacetate: b.p. 134–6°/16 mm. (133°/29 mm.). D_4^{20} 1·1495. $n_D^{20\cdot3}$ 1·44052. Sol. usual org. solvents. Spar. sol. H_2O.

Phenylbenzylhydrazone: pale yellow needles from MeOH. M.p. 67°.

2 : 4-*Dinitrophenylhydrazone*: pale yellowish-brown prisms from AcOEt. M.p. 130–1°.

Scheibler, Sotscheck, Friese, *Ber.*, 1925, **58**, 1961; 1924, **57**, 1443.
Burdick, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 440.
Covert, Connor, Adkins, *J. Am. Chem. Soc.*, 1932, **54**, 1656.
Minné, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 304.

Tetrahydrofurfuryl Alcohol



$C_5H_{10}O_2$ MW, 102

B.p. 177–8°/750 mm., 80–2°/20 mm. D_4^{25} 1·1326. n_D^{25} 1·4505. Absorbs moisture from the air. Solvent for fats, waxes, resins, etc.

Et ether: $C_7H_{14}O_2$. MW, 130. B.p. 152–4°/726 mm., 47–55°/11 mm.

Acetyl: b.p. 192–4°/740 mm., 88–90°/18 mm. D_4^{25} 1·0624. n_D^{25} 1·4350.

Iodoacetyl: b.p. 130°/5 mm.

Propionyl: b.p. 204–7°/756 mm., 85–7°/3 mm. D_4^{20} 1·044.

Butyryl: b.p. 225–7°/759 mm., 102–4°/4 mm. D_0^{20} 1·012.

Valeryl: b.p. 238–40°/756 mm., 97–9°/2 mm. D_0^{20} 0·999.

Pyruvyl: b.p. 110–30°/17 mm. *Semicarbazone*: leaflets. M.p. 184–6°.

Benzoyl: b.p. 300–2°/750 mm., 138–40°/2 mm. D_0^{20} 1·137.

p-*Nitrobenzoyl*: leaflets from EtOH. M.p. 46–8°.

3 : 5-*Dinitrobenzoyl*: small needles from EtOH. M.p. 83–4°.

Hydrogen phthalate: rhombs from Et_2O–pet. ether. M.p. 62–4°.

Hydrogen 3-*nitrophthalate*: cryst. from H_2O. M.p. 75–7°.

p-*Toluenesulphonyl*: m.p. 39–40°.

Phenylurethane: cryst. from pet. ether. M.p. 61°.

Diphenylurethane: plates from MeOH. M.p. 81°.

α-*Naphthylurethane*: needles from pet. ether. M.p. 88–90°.

d-.

B.p. 79°/20 mm. $[\alpha]_D^{20}$ + 2·2°. n_D^{17} 1·4531.

Hydrogen phthalate: rhombs from Et_2O–pet. ether. M.p. 82–3°.

l-.

B.p. 74·5°/14 mm. $[\alpha]_D^{20}$ − 2·2°. n_D^{20} 1·4529.

p-*Nitrobenzoyl*: long needles from Et_2O–pet. ether. M.p. 36–7°.

Hydrogen phthalate: rhombs from Et_2O–pet. ether. M.p. 82–83·5°.

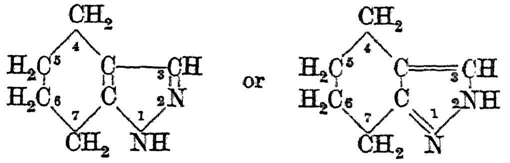
Wienhaus, *Ber.*, 1920, **53**, 1656.
Zanetti, *J. Am. Chem. Soc.*, 1928, **50**, 1821.
du Pont, B.P. 337,296, (*Chem. Abstracts*, 1931, **25**, 1844).
Böhme, B.P. 388,703, (*Chem. Abstracts*, 1933, **27**, 4547).
Hewlett, *Chem. Abstracts*, 1933, **27**, 980.
Burdick, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 441.

Tetrahydrofuroic Acid.
See Tetrahydropyromucic Acid.

Tetrahydrogeraniol.
See 3 : 7-Dimethyloctanol-1.

Tetrahydroiminazolone-2.
See Ethyleneurea.

4 : 5 : 6 : 7-Tetrahydroindazole



$C_7H_{10}N_2$ MW, 122

Cryst. M.p. 84°.

1-*Benzoyl*: prisms from pet. ether. M.p. 58·5–60°. B.p. 208°/13 mm. Sol. most org. solvents.

1-o-*Nitrobenzoyl*: cryst. from EtOH. M.p. 148–9°.

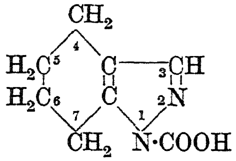
1-m-*Nitrobenzoyl*: needles from EtOH. M.p. 140–2°. Sol. $CHCl_3$, C_6H_6. Mod. sol. EtOH, Et_2O.

2-o-*Nitrobenzoyl*: cryst. from EtOH. M.p. 179–80°. Sol. Me_2CO, $CHCl_3$, C_6H_6. Less sol. EtOH, Et_2O.

2-m-*Nitrobenzoyl*: leaflets from EtOH. M.p. 118–118·5°.

Auwers, *Ann.*, 1927, **453**, 227.
Wallach, *Ann.*, 1903, **329**, 118.

4 : 5 : 6 : 7-Tetrahydroindazole-1-carboxylic Acid



$C_8H_{10}O_2N_2$ MW, 166

Et ester: $C_{10}H_{14}O_2N_2$. MW, 194. B.p. 170°/18 mm. D_4^{20} 1·127. n_{He}^{20} 1·5147.

Chloride: $C_8H_9ON_2Cl$. MW, 184·5. Cryst. powder from C_6H_6. M.p. 162–5°.

Amide: cryst. from AcOH.Aq. M.p. 187°.

Auwers, *Ann.*, 1927, **453**, 230.

4 : 5 : 6 : 7-Tetrahydroindazole-2-carboxylic Acid.

Et ester: b.p. 170°/18 mm. D_4^{20} 1·138. n_{He}^{20} 1·5120.

Amide: cryst. M.p. 186–8°.

Auwers, *Ann.*, 1927, **453**, 230.
Wallach, *Ann.*, 1903, **329**, 117.

4 : 5 : 6 : 7-Tetrahydroindazole-3-carboxylic Acid.

Needles from AcOH. M.p. 254°. Mod. sol. AcOH. Spar. sol. EtOH. Very spar. sol. Et_2O, Me_2CO, C_6H_6.

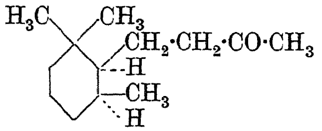
Me ester: 1-o-*nitrobenzoyl*, needles from AcOH. M.p. 193–4°. Sol. hot EtOH, AcOH. Spar. sol. C_6H_6.

Et ester: cryst. from pet. ether. M.p. 106–7°. Sol. most org. solvents.

1-o-*Nitrobenzoyl*: m.p. 148–9°.

Auwers, *Ann.*, 1927, **453**, 232.
Auwers, Ernecke, Conrad, Ottens, *Ann.*, 1929, **469**, 68.

Tetrahydroionone



$C_{13}H_{24}O$ MW, 196

Cis (as shown):

Obtained by PtO_2/H red. of β-ionone. Colourless oil. B.p. 97–8°/2 mm. D_4^{20} 0·9109, D_4^{22} 0·9138. n_D^{20} 1·46720, n_D^{22} 1·4660. $[\alpha]_D - 16°$ in $CHCl_3$.

2 : 4-*Dinitrophenylhydrazone*: cryst. from MeOH. M.p. 120·0–120·5°.

Semicarbazone: cryst. from 95% EtOH. M.p. 179–80° (183–4°).

Phenylsemicarbazone: cryst. from MeOH. M.p. 108–9° (109–10°).

Trans:

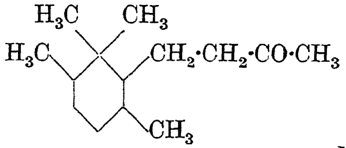
Synthetic. Colourless oil. B.p. 121°/12 mm. D_4^{22} 0·9064. n_D^{22} 1·4634.

Semicarbazone: cryst. from MeOH. M.p. 163°.

Phenylsemicarbazone: needles from MeOH. M.p. 133°.

Prelog, Frick, *Helv. Chim. Acta*, 1948, **31**, 417.
Naves, Ardizio, *Helv. Chim. Acta*, 1949, 32, 206.
Sabotka, Chanley, *J. Am. Chem. Soc.*, 1949, **71**, 4136.

Tetrahydroirone

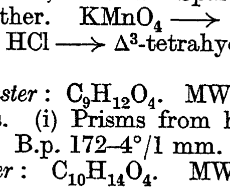


$C_{14}H_{26}O$ MW, 210

Not all the possible isomers of this comp. have been prepared. Those obtained by reduction depend upon the starting material, *i.e.* whether natural or synthetic, and the method of reduction, and their steric relations are not fully understood.

Seidel, Schintz, Ruzicka, *Helv. Chim. Acta*, 1949, **32**, 2102.

Δ¹-Tetrahydroisophthalic Acid (*Cyclohexene*-1 : 3-*dicarboxylic acid*)



$C_8H_{10}O_4$ MW, 170

Cryst. from H_2O. M.p. 197–8° (168°). Very sol. H_2O, EtOH, AcOH. Spar. sol. $CHCl_3$, C_6H_6, pet. ether. $KMnO_4$ ⟶ succinic acid. Hot KOH or HCl ⟶ Δ³-tetrahydroisophthalic acid.

Mono-Me ester: $C_9H_{12}O_4$. MW, 184. Exists in two forms. (i) Prisms from hexane. M.p. 59°. (ii) Oil. B.p. 172–4°/1 mm.

Di-Me ester: $C_{10}H_{14}O_4$. MW, 198. B.p. 134–5°/7 mm.

Mono-Et ester: $C_{10}H_{14}O_4$. MW, 198. Exists in two forms. (i) Prisms from pet. ether. M.p. 44–5°. (ii) Cryst. from pet. ether. M.p. 40–1°. B.p. 169–73°/1 mm.

Di-Et ester: $C_{12}H_{18}O_4$. MW, 226. B.p. 150°/12 mm. D_4^{20} 1·0772. n_D 1·4722.

Amide: prisms from H_2O. M.p. 239°.
Anhydride: plates from C_6H_6–pet. ether. M.p. 78–80°. Sol. warm C_6H_6. Spar. sol. Et_2O.
Monoanilide: needles from AcOH.Aq. M.p. 190–2°.

Kon, Nandi, *J. Chem. Soc.*, 1933, 1631.
Farmer, Richardson, *J. Chem. Soc.*, 1926, 2174.
Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 301.

Δ^3-Tetrahydroisophthalic Acid (*Cyclohexene*-1 : 5-*dicarboxylic acid*).

Cryst. from H_2O. M.p. 243–4°. Sol. boiling formic acid. Spar. sol. boiling H_2O. $KMnO_4$ or HNO_3 ⟶ isophthalic acid.
Di-Me ester: b.p. 140–1°/7 mm.

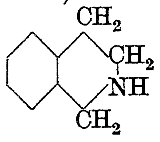
Farmer, Richardson, *J. Chem. Soc.*, 1926, 2176.
Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 306.

Δ^4-Tetrahydroisophthalic Acid (*Cyclohexene*-3 : 5-*dicarboxylic acid*)

Cis:
Needles from H_2O. M.p. 165°. Very sol. hot H_2O. Hot KOH ⟶ Δ^3-tetrahydroisophthalic acid.
Trans:
Cryst. M.p. 225–7°. Spar. sol. H_2O.

Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 310.

1 : 2 : 3 : 4-Tetrahydroisoquinoline (Py-*Tetrahydroisoquinoline*)

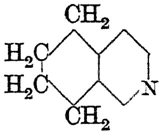


$C_9H_{11}N$ MW, 133

B.p. 232–3°. $D_4^{23\cdot1}$ 1·0642. $n_D^{23\cdot1}$ 1·5798. Spar. sol. H_2O. Heat of comb. C_v 1214 Cal. Reduces $NH_3.AgNO_3$.
B,HCl: plates. M.p. 195–7°.
B_2,H_2PtCl_6: reddish-yellow plates. M.p. 231–2°.
Picrate: yellow needles from EtOH. M.p. 195°.
Picrolonate: m.p. 260° decomp.
N-*Me*: *see* Isokairoline.
N-*Et*: $C_{11}H_{15}N$. MW, 161. Pale yellow oil. B.p. 225–7°. *B,HI*: pale yellow needles from H_2O. M.p. 170°. *Picrate*: yellow needles from EtOH. M.p. 121°.
N-*Propyl*: $C_{12}H_{17}N$. MW, 175. B.p. 259–60°/743 mm.
N-*Isopropyl*: b.p. 256–8°/735 mm.
N-*Butyl*: $C_{13}H_{19}N$. MW, 189. B.p. 272–3°.
N-*Isoamyl*: $C_{14}H_{21}N$. MW, 203. B.p. 276–80°.
N-*Benzyl*: pale yellow oil. B.p. 194–7°/18 mm.
N-*Phenacyl*: yellow needles from EtOH. M.p. 100–1°.
N-*Acetyl*: cryst. from ligroin. M.p. 46°. B.p. 220–5°/70 mm. Sol. usual org. solvents.
N-*Benzoyl*: m.p. 129°. B.p. 245–50°/50 mm.
N-o-*Nitrobenzoyl*: needles from EtOH. M.p. 75–6°. Sol. EtOH, C_6H_6. Spar. sol. pet. ether.
N-*Benzenesulphonyl*: m.p. 154°.

Kondo, Ochiai, *Chem. Abstracts*, 1923, **17**, 3032.
Wedekind, Ney, *Ber.*, 1909, **42**, 2140; 1912, **45**, 1308.
Bamberger, Dieckmann, *Ber.*, 1893, **26**, 1213.
Pictet, Spengler, *Ber.*, 1911, **44**, 2034.
Pictet, D.R.P., 241,425, (*Chem. Zentr.*, 1912, I, 177).
Cromwell, Cram, *J. Am. Chem. Soc.*, 1943, **65**, 305.

5 : 6 : 7 : 8-Tetrahydroisoquinoline (Bz-*Tetrahydroisoquinoline*)

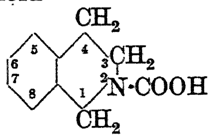


$C_9H_{11}N$ MW, 133

B.p. 218°. D_4^{10} 1·0504. n_D^{10} 1·57263.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

1 : 2 : 3 : 4 - Tetrahydroisoquinoline - 2 - carboxylic Acid



$C_{10}H_{11}O_2N$ MW, 177

Amide: $C_{10}H_{12}ON_2$. MW, 176. Leaflets from H_2O. M.p. 169°. Spar. sol. Et_2O.
Anilide: needles from EtOH. M.p. 144°. Spar. sol. EtOH, Et_2O. Insol. H_2O.

Bamberger, Dieckmann, *Ber.*, 1893, **26**, 1212.

1 : 2 : 3 : 4 - Tetrahydroisoquinoline - 3 - carboxylic Acid.

Cryst. from EtOH.Aq. M.p. 335° (311°) decomp. Mod. sol. warm H_2O. Spar. sol. $CHCl_3$. Insol. EtOH, Et_2O. Heat above m.p. ⟶ 1 : 2 : 3 : 4-tetrahydroisoquinoline.
Et ester: b.p. 120°/1 mm. *Picrate*: yellow cryst. from EtOH. M.p. 204°. *Picrolonate*: yellow needles from EtOH. M.p. 212–13°.

Pictet, Spengler, *Ber.*, 1911, **44**, 2034.
Pictet, D.R.P., 241,425, (*Chem. Zentr.*, 1912, I, 177).
Julian, Karpel, Magnani, Meyer, *J. Am. Chem. Soc.*, 1948, **70**, 182.

5 : 6 : 7 : 8-Tetrahydroisoquinoline-3-carboxylic Acid.

Cryst. from H_2O. M.p. 203–6° (208°). Decarboxylates at 230°.

Scholz, *Helv. Chim. Acta*, 1935, **18**, 930.
Julian, Karpel, Magnani, Meyer, *J. Am. Chem. Soc.*, 1948, **70**, 182.

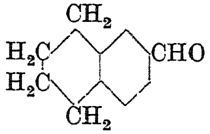
Tetrahydrolaccol.
See under Laccol.

Tetrahydrolepidine.
See 4-Methyltetrahydroquinoline.

Tetrahydrolinalool.
See 3 : 7-Dimethyloctanol-3.

Tetrahydromesitylene.
See 1 : 3 : 5-Trimethylcyclohexene.

5 : 6 : 7 : 8-Tetrahydro-2-naphthaldehyde

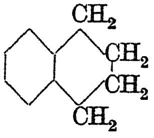


$C_{11}H_{12}O$ MW, 116

B.p. 116–19°/3 mm.
Semicarbazone : m.p. 223–4° decomp.

Newman, Zahm, *J. Am. Chem. Soc.*, 1943, **65**, 1097.

Tetrahydronaphthalene (*Tetralin*)



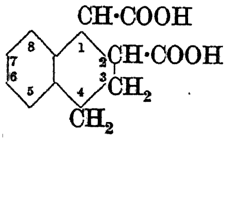
$C_{10}H_{12}$ MW, 132

M.p. — 35°. B.p. 207·3°, 90·8–91·2°/17 mm. D_4^{17} 0·9738. n_D^{17} 1·54529. Sp. heat 0·403 at 18°. Heat of vap. 79·32 cal. per gram. Heat of comb. C_p 1353 Cal. Crit. temp. 789°. Used extensively as solvent for org. substances. Non-toxic.

α-Hydroperoxide : >CH·OOH-α. $C_{10}H_{12}O_2$. MW, 164. Needles from pet. ether. M.p. 56°. $D_4^{78\cdot8}$ 1·0988. $n_D^{78\cdot8}$ 1·53893. Sol. $CHCl_3$.

Graebe, Guye, *Ber.*, 1883, **16**, 3028.
Heaton, *Journal of Oil and Colour Chemists Association*, 1923, **6**, 93.
Inoue, *Chem. Abstracts*, 1924, **18**, 2697.
Gewerkschaft Mathias Stinnes, D.R.P., 610,829, (*Chem. Abstracts*, 1935, **29**, 5866).
I.C.I., B.P. 401,724, (*Chem. Abstracts*, 1934, **28**, 2723).
Musser, Adkins, *J. Am. Chem. Soc.*, 1938, **60**, 664.
Hock, Lang, *Ber.*, 1942, **75**, 313.

1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 2-dicarboxylic Acid



$C_{12}H_{12}O_4$ MW, 220

Needles. M.p. 193° (rapid heat.). Sol. Me_2CO. Mod. sol. EtOH, AcOH, hot H_2O. Spar. sol. C_6H_6.

Anhydride : $C_{12}H_{10}O_3$. MW, 202. Needles from MeOH. M.p. 66–7°. Sol. AcOH. Spar. sol. C_6H_6.

Auwers, Möller, *J. prakt. Chem.*, 1925, **109**, 142.

1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 8-dicarboxylic Acid (1 : 2 : 3 : 4-*Tetrahydronaphthalic acid*).

Prisms. M.p. 196°. Sol. EtOH. Spar. sol. warm H_2O. Almost insol. Et_2O. Above m.p. ⟶ anhydride.

Di-Me ester : $C_{14}H_{16}O_4$. MW, 248. Cryst. M.p. 74°.

Di-Et ester : $C_{16}H_{20}O_4$. MW, 276. M.p. 52°. B.p. 193°/17 mm.

Anhydride : cryst. from C_6H_6. M.p. 119°.

Cassares, Ranedo, *Chem. Abstracts*, 1923, **17**, 3030.
Willstätter, Jacquet, *Ber.*, 1918, **51**, 775.

1 : 2 : 3 : 4-Tetrahydronaphthalene-2 : 3-dicarboxylic Acid.

Cis :

Large prisms from MeOH.Aq. M.p. 194–5°. pK_1 3·98, pK_2 6·47.

Me ester : prisms from ligroin. M.p. 68–68·5°.

Anhydride : prisms from Et_2O. M.p. 184°. Mod. sol. EtOH, $CHCl_3$. Spar. sol. Et_2O. Insol. cold H_2O.

Trans :

d-.

Prisms from Me_2CO–C_6H_6. M.p. 182–3°. $[\alpha]_D^{16}$ + 85·5° in $CHCl_3$.

l-.

Prisms from Me_2CO–C_6H_6. M.p. 182–3°. $[\alpha]_D^{16}$ — 85·0° in $CHCl_3$.

dl-.

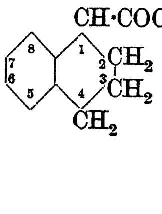
Prismatic plates from EtOH.Aq. M.p. 226–7°. pK_1 4·00, pK_2 5·70.

Me ester : hexagonal prisms from ligroin. M.p. 44·5–45°.

Baeyer, Perkin, *Ber.*, 1884, **17**, 450.
Haworth, Slinger, *J. Chem. Soc.*, 1940, 1324.

Tetrahydronaphthalic Acid.
See Tetrahydronaphthalene-1 : 8-dicarboxylic Acid.

1 : 2 : 3 : 4-Tetrahydro-1-naphthoic Acid (ac-*Tetrahydro-α-naphthoic acid*)



$C_{11}H_{12}O_2$ MW, 176

d-.

Cryst. from pet. ether. M.p. 49–50°. $[\alpha]_D$ + 14·01° in $CHCl_3$.

l-.

Plates from pet. ether. M.p. 52·5°. $[\alpha]_D$ − 52·34° in C_6H_6, − 15·95° in $CHCl_3$.

dl-.

Prisms from AcOEt. M.p. 85°. Very sol. most org. solvents. Mod. sol. hot H_2O. Sol. 1052 parts cold H_2O. $k = 4{\cdot}45 \times 10^{-5}$ at 25°.

Et ester: $C_{13}H_{16}O_2$. MW, 204. B.p. 279°/749 mm.

l-*Menthyl ester*: b.p. 207°/10 mm. $[\alpha]_D^{20}$ − 47·57° in EtOH.

Amide: $C_{11}H_{13}ON$. MW, 175. Needles from EtOH. M.p. 116°.

Pickard, Yates, *J. Chem. Soc.*, 1906, **89**, 1102.

Kay, Morton, *J. Chem. Soc.*, 1914, **105**, 1571.

5 : 6 : 7 : 8-Tetrahydro-1-naphthoic Acid (ar-*Tetrahydro-α-naphthoic acid*).

Prisms from H_2O. M.p. 128°. Very sol. EtOH. Sol. hot H_2O. Spar. sol. cold H_2O.

Et ester: b.p. 156–9°/12 mm.

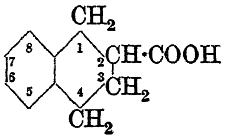
Nitrile: $C_{11}H_{11}N$. MW, 157. B.p. 277–9°/721 mm.

Amide: needles from H_2O. M.p. 182°. Sol. boiling H_2O and most org. solvents.

Bamberger, Bordt, *Ber.*, 1889, **22**, 628.

I.G., F.P., 649,626, (*Chem. Abstracts*, 1929, **23**, 2986).

1 : 2 : 3 : 4-Tetrahydro-2-naphthoic Acid (ac-*Tetrahydro-β-naphthoic acid*)



$C_{11}H_{12}O_2$ MW, 176

d-.

Prisms from AcOH.Aq. M.p. 99°. $[\alpha]_D$ + 40·35° in $CHCl_3$.

l-.

Prismatic needles from pet. ether. M.p. 99°. $[\alpha]_D$ − 51·82° in $CHCl_3$.

dl-.

Needles from hot EtOH.Aq. M.p. 96°. Sol. 1661 parts H_2O at 14°.

l-*Menthyl ester*: b.p. 218°/11 mm. $[\alpha]_D^{20}$ − 53·0° in C_6H_6.

Pickard, Yates, *J. Chem. Soc.*, 1906, **89**, 1103.

Baeyer, Besemfelder, *Ann.*, 1891, **266**, 198.

5 : 6 : 7 : 8-Tetrahydro-2-naphthoic Acid (ar-*Tetrahydro-β-naphthoic acid*).

Needles from EtOH. M.p. 154° (144°). B.p. 216°/14 mm. Insol. cold pet. ether.

Chloride: $C_{11}H_{11}OCl$. MW, 194·5. B.p. 162°/13 mm.

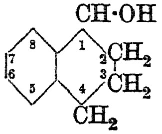
Amide: $C_{11}H_{13}ON$. MW, 175. Leaflets from EtOH–pet. ether. M.p. 137–8°.

Anilide: needles from EtOH. M.p. 147°.

v. Braun, Kirschbaum, Schumann, *Ber.*, 1920, **53**, 1161.

Coulson, *J. Chem. Soc.*, 1935, 80.

1 : 2 : 3 : 4-Tetrahydro-1-naphthol (ac-*Tetrahydro-α-naphthol*, 1-*hydroxy*-1 : 2 : 3 : 4-*tetrahydronaphthalene*, 1-*hydroxytetralin*)



$C_{10}H_{12}O$ MW, 148

d-.

Pale yellow oil. B.p. 155–60°/25 mm. $[\alpha]_D$ + 28·2° in $CHCl_3$.

dl-.

Thick oil. B.p. 264°/716 mm., 176·5–178°/53 mm., 132–4°/12–13 mm. D^{17} 1·0896. n_D^{17} 1·5671. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. H_2O.

Acetyl: b.p. 169°/34 mm., 105–10°/2 mm.

Phenylurethane: needles from ligroin. M.p. 121–2°.

Criegee, *Ann.*, 1930, **481**, 292.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

Strauss, Rohrbacher, *Ber.*, 1921, **54**, 57.

Bamberger, Lodter, *Ber.*, 1890, **23**, 197.

5 : 6 : 7 : 8-Tetrahydro-1-naphthol (ar-*Tetrahydro-α-naphthol*, 5-*hydroxy*-1 : 2 : 3 : 4-*tetrahydronaphthalene*, 5-*hydroxytetralin*, 1-*tetralol*).

Needles with strong phenolic odour. M.p. 68·5–69°. B.p. 264·5–265°. Very sol. most org. solvents. Less sol. hot H_2O.

Et ether: $C_{12}H_{16}O$. MW, 176. B.p. 259°/705 mm.

Acetyl: cryst. M.p. 73–5°.

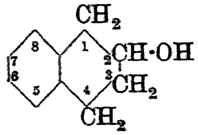
Benzoyl: m.p. about 46°.

Jacobsen, Turnbull, *Ber.*, 1898, **31**, 897.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

I.G., F.P., 644,408, (*Chem. Abstracts*, 1930, **24**, 862); U.S.P., 1,858,627, (*Chem. Abstracts*, 1932, **26**, 3808).

1 : 2 : 3 : 4-Tetrahydro-2-naphthol (ac-*Tetrahydro-β-naphthol*, 2-*hydroxy*-1 : 2 : 3 : 4-*tetrahydronaphthalene*, 2-*hydroxytetralin*)



$C_{10}H_{12}O$ MW, 148

d-.
Needles from pet. ether. M.p. 50°. B.p. 141°/17 mm. $[\alpha]_D^{20}$ + 70·3° in $CHCl_3$.
Valeryl: b.p. 169°/11 mm. D_4^{16} 1·0317.
Pelargonyl: b.p. 195–7°/5 mm. D_4^{18} 0·9821.
Lauryl: cryst. M.p. 35–6°. B.p. 218°/3 mm. D_4^{17} 0·9677.
Phenylurethane: m.p. 119°.

l-.
Needles from pet. ether. M.p. 50°. B.p. 141°/17 mm. D_4^{61} 1·0589. $[\alpha]_D^{20}$ − 67·1° in $CHCl_3$.
Acetyl: b.p. 149°/18 mm. D_4^{14} 1·0926.
Propionyl: b.p. 158–9°/18 mm. D_4^{16} 1·0675.

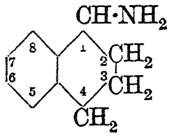
dl-.
Viscous oil. B.p. 264°/716 mm., 172·6–173·5°/53 mm., 144·5–146·5°/20 mm. D^{17} 1·0715. n_D^{17} 1·5523. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. H_2O. Turns brown in air.
Acetyl: b.p. 169°/34 mm.
Benzoyl: leaflets from EtOH. B.p. 254–5°/40 mm. Sol. hot EtOH, cold $CHCl_3$, C_6H_6.
Phenylurethane: needles. M.p. 99°.

Bamberger, Lodter, *Ber.*, 1890, **23**, 197.
Pickard, Kenyon, *J. Chem. Soc.*, 1912, **101**, 1431.
Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

5 : 6 : 7 : 8-Tetrahydro-2-naphthol (ar-*Tetrahydro-β-naphthol*, 6-*hydroxy*-1 : 2 : 3 : 4-*tetrahydronaphthalene*, 6-*hydroxytetralin*, 2-*tetralol*).
Needles from ligroin. M.p. 61·5–62·5° (58°). B.p. 275°/705 mm., 146°/13 mm. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, hot ligroin. Spar. sol. H_2O. No col. with $FeCl_3$.
Me ether: $C_{11}H_{14}O$. MW, 162. B.p. 129–31°/11 mm.
Et ether: $C_{12}H_{16}O$. MW, 176. Oil with pleasant odour. B.p. 132–3°/15 mm., 129°/11 mm. D^{20} 1·008.
Acetyl: viscous oil. B.p. 158°/14 mm.
Benzoyl: prisms. M.p. 96°. B.p. 220–2°/10 mm.
p-*Nitrobenzoyl*: pale yellow leaflets from MeOH. M.p. 113° (106·5°).
Cinnamoyl: m.p. 77·5°.
Diphenylurethane: m.p. 114°.

Schroeter, Schrauth, D.R.P., 299,603, (*Chem. Zentr.*, 1919, IV, 618).
Schroeter *et al.*, *Ann.*, 1922, **426**, 119.
Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.
Thoms, Kross, *Arch. Pharm.*, 1927, **265**, 336.
I.G., F.P., 644,408, (*Chem. Abstracts*, 1930, **24**, 862).
Stork, *J. Am. Chem. Soc.*, 1947, **69**, 578.

1 : 2 : 3 : 4-Tetrahydro-1-naphthylamine (ac-*Tetrahydro-α-naphthylamine*)



$C_{10}H_{13}N$ MW, 147

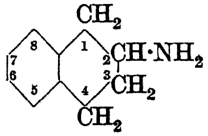
Oil with ammoniacal odour. B.p. 246·5°/714 mm. Sol. most org. solvents. Mod. sol. cold H_2O. Strong base.
B,HNO₂: needles from H_2O. M.p. 138–9°.
B_2,H_2PtCl_6: orange-yellow prisms + $2H_2O$. M.p. 140°, anhyd. 190°.
N-*Acetyl*: needles from EtOH.Aq. M.p. 148–9°. Sol. EtOH, AcOH. Spar. sol. cold H_2O.
N-2 : 4-*Dinitrophenyl*: golden-yellow leaflets from EtOH. M.p. 121°.

Bamberger, Bamman, *Ber.*, 1889, **22**, 964.
Green, Rowe, *J. Chem. Soc.*, 1918, **113**, 957.
Komatsu, Amatatsu, *Chem. Abstracts*, 1931, **25**, 500.

5 : 6 : 7 : 8-Tetrahydro-1-naphthylamine (ar-*Tetrahydro-α-naphthylamine*).
Oil. B.p. 275°, 233–5°/280 mm. D^{16} 1·0625. $n_D^{23·1}$ 1·58964. Weak base. Reduces warm alc. Ag sols.
N-*Me*: $C_{11}H_{15}N$. MW, 161. Pale yellow liq. B.p. 150–2°/12 mm. *Picrate*: reddish-yellow cryst. powder. M.p. 174°.
N-*Di-Me*: $C_{12}H_{17}N$. MW, 175. B.p. 261–2°/721 mm., 131–131·5°/16 mm. Reduces Ag salts. *Methiodide*: prisms from H_2O. M.p. 164·5°. Sol. EtOH. Spar. sol. H_2O.
N-*Et*: $C_{12}H_{17}N$. MW, 175. B.p. 286–7°/717 mm. Sol. most org. solvents. Spar. sol. H_2O.
N-2 : 4-*Dinitrophenyl*: red leaflets from EtOH. M.p. 134°.
N-*Acetyl*: needles. M.p. 158°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

Bamberger, Althausse, *Ber.*, 1888, **21**, 1789.
Bamberger, Helwig, *Ber.*, 1889, **22**, 1315.
Bayer, D.R.P., 305,347, (*Chem. Zentr.*, 1918, I, 977).
Cassella, D.R.P., 479,401, (*Chem. Abstracts*, 1929, **23**, 4710).
I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, **28**, 1059); Swiss P., 127,524. (*Chem. Abstracts*, 1929, **23**, 1143).

1 : 2 : 3 : 4 - Tetrahydro - 2 - naphthyl - amine (ac-*Tetrahydro-β-naphthylamine*)



$C_{10}H_{13}N$ MW, 147

d-.

B,HCl: needles. M.p. 243–5°. $[\alpha]_D^{12}$ + 71·9° in H_2O.

N-*Acetyl*: needles from C_6H_6. M.p. 104–6°. $[\alpha]_D^{16\cdot5}$ + 36·9° in C_6H_6.

N-*Benzoyl*: needles from Me_2CO. M.p. 155–7°. $[\alpha]_D^{19}$ + 58° in Me_2CO.

l-.

B,HCl: cryst. from Me_2CO. M.p. 243–5°. $[\alpha]_D^{16}$ − 69·7° in H_2O.

dl-.

B.p. 249°/710 mm. decomp., 140–140·5°/20 mm., 118·5°/8 mm. $D_4^{22\cdot2}$ 1·0295. $n_D^{22\cdot2}$ 1·56039. Sol. most org. solvents. Spar. sol. cold H_2O. Turns brown in air. Absorbs CO_2.

B,HCl: plates from H_2O. M.p. 237°.

N-*Me*: $C_{11}H_{15}N$. MW, 161. B.p. 118–119·8°/9 mm. D_4^{20} 1·024. Sol. most org. solvents. Spar. sol. H_2O. N-*Acetyl*: yellowish syrup. B.p. 190–210°/17 mm. Sol. most org. solvents. Spar. sol. H_2O.

N-*Di-Me*: $C_{12}H_{17}N$. MW, 175. Oil with strong violet fluor. B.p. 132·3–133·3°/11 mm. Sol. most org. solvents. Spar. sol. H_2O. *B,HCl*: leaflets. M.p. 214–15° decomp. *Methiodide*: cryst. from EtOH. M.p. 228°.

N-*Et*: $C_{12}H_{17}N$. MW, 175. B.p. 267°/724 mm., 153°/23 mm. Volatile in steam. *B,HCl*: needles. M.p. 223·5°. *Picrate*: orange-red needles from H_2O. M.p. 183·5°. N-*Acetyl*: b.p. 328°/718 mm. Sol. most org. solvents. Insol. H_2O.

N-*Formyl*: needles from C_6H_6. M.p. 61°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. cold H_2O, ligroin.

N-*Acetyl*: needles from C_6H_6. M.p. 107·5°. Sol. $CHCl_3$, C_6H_6. Mod. sol. hot H_2O, EtOH. Spar. sol. Et_2O. Insol. ligroin.

N-*Benzoyl*: needles from C_6H_6. M.p. 150–1°. Sol. hot C_6H_6. Spar. sol. hot H_2O.

Bamberger, Müller, *Ber.*, 1889, **22**, 1301.
Waser, *Ber.*, 1916, **49**, 1203.
Waser, Möllering, *Organic Syntheses*, Collective Vol., I, 486.
I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, **28**, 1059).

5 : 6 : 7 : 8-Tetrahydro-2-naphthylamine (ar-*Tetrahydro-β-naphthylamine*).

Needles from ligroin. M.p. 38°. B.p. 275–7°/713 mm. Sol. most org. solvents.

N-*Me*: b.p. 267·5°/210 mm. Sol. most org. solvents. Spar. sol. H_2O.

N-*Di-Me*: b.p. 287°/718 mm., 168°/23 mm. Reduces Ag salts.

N-*Et*: b.p. 291–3°. Sol. most org. solvents. Spar. sol. H_2O. Reduces hot $NH_3.AgNO_3$. *B,HCl*: needles. M.p. 173·5°.

N-*Di-Et*: b.p. 298°/709 mm., 167°/16 mm. Sol. most org. solvents. Spar. sol. H_2O.

N-*Acetyl*: needles. M.p. 107°. Sol. EtOH.

N-*Benzoyl*: m.p. 167°.

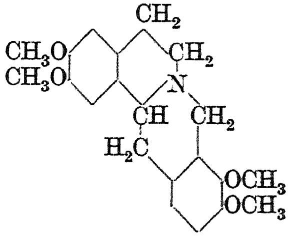
Picrate: m.p. 204°.

Bamberger, Müller, *Ber.*, 1889, **22**, 1304.
Bamberger, Kitschelt, *Ber.*, 1890, **23**, 882.
Cassella, D.R.P., 479,401, (*Chem. Abstracts*, 1929, **23**, 4710).
I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, **28**, 1059).

Δ^3-Tetrahydronicotinic Acid.

See Guvacine.

Tetrahydropalmatine



$C_{21}H_{25}O_4N$ MW, 355

d-.

Alkaloid from buds of *Corydalis cava*. M.p. 143°. $[\alpha]_D^{20}$ + 291° in EtOH. Turns yellow in air.

B,HCl: m.p. 266°.

l-.

M.p. 141–2°. $[\alpha]_D^{14}$ − 290·8° in EtOH.

Hydrate: m.p. 115–16°.

dl-.

B,HCl: needles from H_2O. M.p. 215°.

B,HI: orange-yellow needles. M.p. 241° decomp.

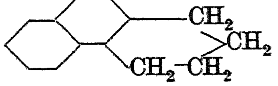
B,HAuCl₄: plates from EtOH.Aq. M.p. 201°.

B₂,H₂PtCl₆: orange cryst. powder. M.p. 228° decomp.

Methiodide: exists in two forms. (i) Cryst. from MeOH. M.p. 230°. (ii) M.p. 266° decomp.

Späth, Mosettig, Tröthandl, *Ber.*, 1923, **56**, 875.
Späth, Mosettig, *Ber.*, 1926, **59**, 1496.
Späth, Leithe, *Ber.*, 1930, **63**, 3007.
Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 553.

1 : 2 : 3 : 4 - Tetrahydrophenanthrene (*Tetanthrene*)



$C_{14}H_{14}$ MW, 182

Cryst. from MeOH. M.p. 33–4°. B.p. 173°/11 mm. D_4^{40} 1·0601. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6, ligroin. Sol. 15 parts MeOH.

Picrate: reddish-yellow needles from EtOH. M.p. 111°.

Schroeter, Müller, Huang, *Ber.*, 1929, **62**, 652.

Tetrahydrophenetole.
See under Cyclohexenol.

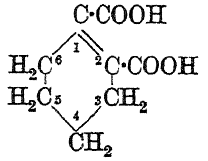
Tetrahydrophenol.
See Cyclohexenol.

Tetrahydrophenylacetaldehyde.
See Cyclohexenylacetaldehyde.

Tetrahydrophenylacetone.
See Cyclohexenylacetone.

Tetrahydrophenylpropionic Acid.
See Cyclohexenylpropionic Acid.

Δ¹-Tetrahydrophthalic Acid (*Cyclohexene-1 : 2-dicarboxylic acid*)



$C_8H_{10}O_4$ MW, 170

Plates from H_2O. M.p. 126° (Heat ⟶ anhydride before melting). Boil with alkalis ⟶ Δ²-tetrahydrophthalic acid. Alk. $KMnO_4$ ⟶ adipic acid.

Di-Et ester: $C_{12}H_{18}O_4$. MW, 226. B.p. 160°/14 mm., 147°/10 mm. $D_4^{19.2}$ 1·0803. $n_{He}^{19.2}$ 1·47466.

Monoamide: $C_8H_{11}O_3N$. MW, 169. Needles from EtOH. Decomp. at 170° ⟶ Δ¹-tetrahydrophthalimide.

Anhydride: $C_8H_8O_3$. MW, 152. Plates from Et_2O. M.p. 74°. Sol. Et_2O.

Imide: $C_8H_9O_2N$. MW, 151. Cryst. from EtOH, Et_2O, or ligroin. M.p. 169–70°. Sol. Me_2CO, $CHCl_3$.

Anil: leaflets or needles from AcOH. M.p. 137°. Sol. EtOH, AcOH, Me_2CO.

Monoanilide: cryst. M.p. 155° (rapid heat.). Sol. EtOH, Me_2CO. Spar. sol. C_6H_6. $FeCl_3$ ⟶ pale green col.

Kon, Nandi, *J. Chem. Soc.*, 1933, 1633.
Hückel, Lampert, *Ber.*, 1934, **67**, 1812.
Baeyer, *Ann.*, 1890, **258**, 203.
Küster, *Z. physiol. Chem.*, 1908, **55**, 520.
Alder, Backendorf, *Ber.*, 1938, **71**, 2206.

Δ²-Tetrahydrophthalic Acid (*Cyclohexene-1 : 6-dicarboxylic acid*).

Prisms. M.p. 215° (rapid heat.). Sol. 114 parts H_2O at 10°. k (first) = 7.4×10^{-5} at 25°; (second) = 3.2×10^{-7} at 100°. Above 220° ⟶ anhydride.

Di-Et ester: b.p. 155°/12 mm. D_4^{20} 1·0760. n_D 1·4700.

Dichloride: $C_8H_8O_2Cl_2$. MW, 207. Yellow oil. B.p. 129°/14 mm.

Anhydride: prisms from Et_2O. M.p. 78–9°.

Imide: m.p. 172–3°. Sol. EtOH, Et_2O, $CHCl_3$, Me_2CO. Insol. cold H_2O.

Kon, Nandi, *J. Chem. Soc.*, 1933, 1633.
Küster, *Z. physiol. Chem.*, 1908, **55**, 520.
Baeyer, *Ann.*, 1890, **258**, 199.
Kaufmann, Voss, *Ber.*, 1923, **56**, 2513.

Δ³-Tetrahydrophthalic Acid (*Cyclohexene-3 : 4-dicarboxylic acid*).

Free acid not isolated. $k = 5.81 \times 10^{-4}$ at 25°.

Anhydride: leaflets. M.p. 70°. Sol. EtOH. Spar. sol. H_2O.

p-*Methoxyanil*: leaflets. M.p. 88°.

Abati, de Bernardinis, *Gazz. chim. ital.*, 1906, 36, ii, 824.

Δ⁴-Tetrahydrophthalic Acid (*Cyclohexene-4 : 5-dicarboxylic acid*).

Cis:

Prisms from H_2O. M.p. 174° (166°). Sol. 108 parts H_2O at 6°.

Anhydride: cryst. from ligroin. M.p. 103–4° (58–9°). Sol. usual org. solvents. Spar. sol. ligroin, pet. ether.

Trans:

d-.

Powder. M.p. 165°. $[\alpha]_D^{25}$ + 115·2°.

Anhydride: leaflets. M.p. 128°. Sol. EtOH, C_6H_6. $[\alpha]_D^{25}$ + 6·6° in EtOH.

l-.

Powder. M.p. 167°. Mod. sol. EtOH. $[\alpha]_D^{25}$ − 97·4°.

dl-.

Leaflets from H_2O. M.p. 215–18°. Sol. 690 parts H_2O at 6°.

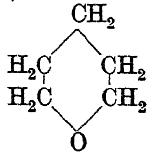
Di-Me ester: $C_{10}H_{14}O_4$. MW, 198. M.p. 39–40°.

Anhydride: cryst. from C_6H_6–ligroin. M.p. 130° (141°).

Diels, Alder, *Ber.*, 1929, **62**, 2087; *Ann.*, 1928, **460**, 113.
Abati, de Horatio, *Gazz. chim. ital.*, 1909, **39**, i, 558.
Baeyer, *Ann.*, 1892, **269**, 203.
I.G., U.S.P., 1,944,731, (*Chem. Abstracts*, 1934, **28**, 2016).

1 : 4 : 5 : 6-Tetrahydro-α-picoline.
See 6-Methyl-1 : 2 : 3 : 4-tetrahydropyridine.

Tetrahydropyran (*Pentamethylene oxide, pyran tetrahydride*)

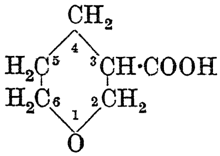


$C_5H_{10}O$ MW, 86

Liq. with characteristic odour. B.p. 88°/760 mm. $D_0^{18.5}$ 0·883, D_4^{15} 0·8855. $n_D^{18.5}$ 1·4195. Volatile in steam.

Clarke, *J. Chem. Soc.*, 1912, **101**, 1802.
Paul, *Bull. soc. chim.*, 1933, **53**, 1493.
Allen, Hibbert, *J. Am. Chem. Soc.*, 1934, **56**, 1398.
Andrus, Johnson, *Organic Syntheses*, 1943, XXIII, 90.

Tetrahydropyran-3-carboxylic Acid



$C_6H_{10}O_3$ MW, 130

Et ester: $C_8H_{14}O_3$. MW, 158. B.p. 101–3°/19–20 mm. D_4^{21} 1·0419. n_D^{21} 1·443.

Nitrile: C_6H_9ON. MW, 111. B.p. 77°/15 mm. D_4^{17} 1·0171. n_D^{17} 1·4428.

Sherlin *et al.*, *Chem. Abstracts*, 1938, **32**, 5398.

Tetrahydropyran-4-carboxylic Acid.

Cryst. from Et_2O. M.p. 87°. B.p. 145–50°/18 mm. Sol. H_2O. Spar. sol. pet. ether.

Me ester: $C_7H_{12}O_3$. MW, 144. Liq. with pleasant odour. B.p. 80·5–81°/16 mm.

Et ester: $C_8H_{14}O_3$. MW, 158. B.p. 82·5°/12 mm.

Chloride: $C_6H_9O_2Cl$. MW, 148·5. B.p. 85–6°/16 mm.

Amide: $C_6H_{11}O_2N$. MW, 129. Plates from EtOH. M.p. 179°.

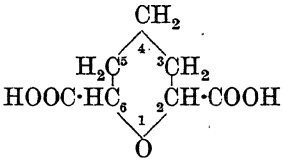
Nitrile: C_6H_9ON. MW, 111. B.p. 82–3°/10 mm. Very sol. H_2O.

Anilide: plates from EtOH. M.p. 163°. Almost insol. Et_2O.

Gibson, Johnson, *J. Chem. Soc.*, 1930, 2527.

v. Braun, Kohler, *Ber.*, 1917, **50**, 1658.

Tetrahydropyran-2 : 6-dicarboxylic Acid



$C_7H_{10}O_5$ MW, 174

Cis-meso:

M.p. 193°.

Di-Me ester: m.p. 54°. B.p. 156°/8 mm.

Anhydride: m.p. 71°.

Diamide: m.p. 263°.

Czornodola, *Chem. Abstracts*, 1937, **31**, 1807.

Tetrahydropyran-4 : 4-dicarboxylic Acid.

Leaflets from Et_2O–pet. ether. M.p. 172–3°. Sol. Et_2O. Spar. sol. pet. ether. Above m.p. ⟶ tetrahydropyran-4-carboxylic acid.

Di-Et ester: $C_{11}H_{18}O_5$. MW, 230. B.p. 152–5°/21 mm., 134–5°/12 mm.

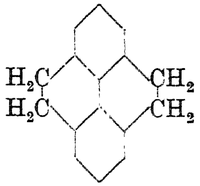
Mononitrile: $C_7H_9O_3N$. MW, 155. Prisms from H_2O. M.p. 160–2°. Very sol. hot H_2O.

Et ester: $C_9H_{13}O_3N$. MW, 183. B.p. 125°/16 mm. *Amide*: $C_7H_{10}O_2N_2$. MW, 154. Plates from EtOH. M.p. 158°.

Gibson, Johnson, *J. Chem. Soc.*, 1930, 2527.

v. Braun, Kohler, *Ber.*, 1917, **50**, 1658.

1 : 2 : 6 : 7-Tetrahydropyrene



$C_{16}H_{14}$ MW, 206

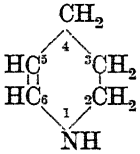
Colourless flakes from EtOH. M.p. 138°. Very sol. pet. ether, C_6H_6, Et_2O.

Coulson, *J. Chem. Soc.*, 1937, 1298.

Tetrahydropyrethrolone.

See under Pyrethrolone.

1 : 2 : 3 : 4-Tetrahydropyridine (Δ^2-*Piperideine*)



C_5H_9N MW, 83

Decomp. on boiling with formation of a dimer.

B,HCl: m.p. 230°.

B,HBr: m.p. 178°.

Acetyl: b.p. 219·5–220·5°. $D_4^{16\cdot5}$ 1·0531. Sol. usual org. solvents.

Dimer: cryst. M.p. 61°. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Heat of comb. C_v 1532·7 Cal. Reduces Ag salts.

Lellmann, Schwaderer, *Ber.*, 1889, **22**, 1320.

Wolffenstein, *Ber.*, 1892, **25**, 2782.

Paal, Hubaleck, *Ber.*, 1901, **34**, 2761.

1 : 2 : 3 : 6-Tetrahydropyridine (Δ^3-*Piperideine*).

Free base polymerises readily.

B,HCl: m.p. 188–9°.

B,HAuCl₄: yellow plates from dil. HCl. M.p. 141–2° decomp.

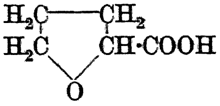
B₂,H₂PtCl₆: yellow plates from alcoholic HCl. M.p. 187–8° decomp.

Renshaw, Conn, *J. Am. Chem. Soc.*, 1938, **60**, 745.

Tetrahydropyridine-carboxylic Acid.

See Guvacine *and* Baikiain.

Tetrahydropyromucic Acid (*Tetrahydrofuran-2-carboxylic acid, tetrahydro-β-furoic acid*)



$C_5H_8O_3$ MW, 116

Cryst. M.p. 21°. B.p. 145°/25 mm., 131°/14 mm. D_{20}^{20} 1·1933. n_D^{19} 1·4585. $k = 1{\cdot}4 \times 10^{-4}$ at 25°.

Et ester: $C_7H_{12}O_3$. MW, 144. Liq. with pleasant fruity odour. B.p. 82°/11 mm. D_{20}^{20} 1·0792. n_D^{18} 1·4445. Sol. EtOH, Et_2O. Insol. H_2O.

p-*Phenylphenacyl ester*: cryst. from EtOH.Aq. M.p. 100·5°.

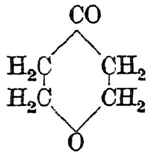
Amide: $C_5H_9O_2N$. MW, 115. Leaflets from Et_2O. M.p. 80°. B.p. 135–40°/20 mm. Sol. H_2O, $CHCl_3$. Spar. sol. Et_2O.

Nitrile: C_5H_7ON. MW, 97. B.p. 80–2°/23 mm. n_D^{25} 1·4351.

Wienhaus, Sorge, *Ber.*, 1913, **46**, 1929.
Kaufmann, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 3041.
Williams, *Ber.*, 1927, **60**, 2512.

Tetrahydro-α-pyrone.
See δ-Valerolactone.

Tetrahydro-γ-pyrone



$C_5H_8O_2$ MW, 100

B.p. 163–6°/742 mm., 67–9°/18 mm. Misc. with H_2O. $D^{24\cdot5}$ 1·0795. $n_D^{24\cdot5}$ 1·4529.

Oxime: cryst. from EtOH. M.p. 87–8°.

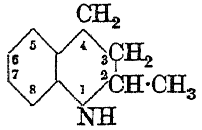
p-*Nitrophenylhydrazone*: orange-brown needles from EtOH. M.p. 186°.

Phenylsemicarbazone: needles from EtOH.Aq. M.p. 169°.

Borsche, *Ber.*, 1915, **48**, 683.
Borsche, Thiele, *Ber.*, 1923, **56**, 2012.
Cornubert, Robinet, *Bull. soc. chim.*, 1933, **53**, 565.
Cawley, Plant, *J. Chem. Soc.*, 1938, 1216.

Tetrahydropyrrole.
See Pyrrolidine.

1 : 2 : 3 : 4-Tetrahydroquinaldine (2-*Methyl*-1 : 2 : 3 : 4-*tetrahydroquinoline*)



$C_{10}H_{13}N$ MW, 147

d-.

Oil. $[\alpha]_D^{20}$ + 58·1°.

B,HCl: plates + H_2O from EtOH. M.p. 196·5–197·5°. $[\alpha]_D^{21\cdot4}$ + 66·1° in H_2O.

N-*Et*: $C_{12}H_{17}N$. MW, 175. B.p. 256°. D_4^{20} 0·9942. $[\alpha]_D^{20}$ + 12·1°.

N-*Benzoyl*: cryst. $[\alpha]_D^{18}$ − 247° in C_6H_6.

l-.

B.p. 158–9°/59 mm. $D_4^{14\cdot5}$ 1·0207. n_D^{24} 1·5705. Misc. with usual org. solvents.

B,HCl: plates + H_2O from EtOH. M.p. 196·5–197·5°. $[\alpha]_D^{19}$ − 60·4° in H_2O.

N-*Benzoyl*: cryst. from EtOH. M.p. 117·5–118°. Sol. C_6H_6. Mod. sol. EtOH. $[\alpha]_D^{25}$ + 324° in C_6H_6.

N-o-*Nitrobenzoyl*: yellow needles from EtOH. M.p. 110°. Sol. EtOH, Me_2CO, AcOH, C_6H_6. $[\alpha]_D^{20}$ + 17·1° in EtOH.

N-m-*Nitrobenzoyl*: needles from EtOH. M.p. 97°. $[\alpha]_D^{20}$ + 241·6° in EtOH.

N-p-*Nitrobenzoyl*: yellowish cryst. from C_6H_6–pet. ether. M.p. 110°. $[\alpha]_D^{20}$ + 369·6° in EtOH.

N-p-*Toluenesulphonyl*: cryst. from EtOH. M.p. 109°. $[\alpha]_D^{20}$ − 137° in EtOH.

Picrate: yellow plates or needles from EtOH. M.p. 148–50°. $[\alpha]_D^{20}$ − 33·0° in EtOH.

dl-.

B.p. 246–8°/709 mm., 115–16°/12 mm. D_4^{18} 1·0208. n_D^{22} 1·5727. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Heat of comb. C_v 1380·6 Cal.

B,HCl: m.p. 190° (128–30°).

N-*Me*: $C_{11}H_{15}N$. MW, 161. B.p. 247–8°, 144°/28 mm. n_D^{19} 1·5678. Turns brown in air. *Methiodide*: needles from EtOH. M.p. 205°.

N-*Et*: b.p. 256°.

N-*Benzoyl*: cryst. from AcOEt. M.p. 119·2° (116°). Sol. most org. solvents.

N-m-*Nitrobenzoyl*: pale yellow leaflets. M.p. 114°.

Picrate: prisms from EtOH. M.p. 153–4°.

Pope, Peachey, *J. Chem. Soc.*, 1899, **75**, 1082.
Pope, Winmill, *J. Chem. Soc.*, 1912, **101**, 2311.
v. Braun, Gmelin, Schultheiss, *Ber.*, 1923, **56**, 1344.
Tröger, Ungar, *J. prakt. Chem.*, 1926, **112**, 251.

5 : 6 : 7 : 8-Tetrahydroquinaldine (2-*Methyl*-5 : 6 : 7 : 8-*tetrahydroquinoline*).

B.p. 225°/762 mm., 101–4°/12 mm. D^{16} 1·0000. n_D^{23} 1·5310.

B,HCl: m.p. 164°.

B_2,H_2PtCl_6: m.p. 198°.

Methiodide: m.p. 118°. Hygroscopic.

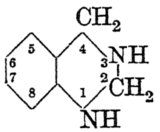
Picrate: lemon-yellow cryst. M.p. 157° (154°).

Picrolonate: yellow prisms. M.p. 220°.

v. Braun, Gmelin, Schultheiss, *Ber.*, 1923, **56**, 1344.
Tröger, Ungar, *J. prakt. Chem.*, 1926, **112**, 251.
Basu, *Ann.*, 1934, **512**, 131.

Tetrahydroquinaldinic Acid.
See Tetrahydroquinoline-2-carboxylic Acid.

1 : 2 : 3 : 4-Tetrahydroquinazoline



$C_8H_{10}N_2$ MW, 134

Leaflets from C_6H_6–ligroin, m.p. 78–9°: cryst. + H_2O from H_2O, m.p. 49–51°.

B,HCl: needles from EtOH. M.p. 193–5°.

B_2,H_2PtCl_6: orange-yellow needles. Does not melt below 270°.

3-N-*Allyl*: oil. B.p. 270–2°.

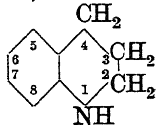
3-N-*Phenyl*: needles from EtOH. M.p. 119°.

3-N-o-*Tolyl*: cryst. from Et_2O. M.p. 140°.

3-N-p-*Tolyl*: needles from EtOH. M.p. 127°.

Busch, Dietz, *J. prakt. Chem.*, 1896, **53**, 418.
Busch, *J. prakt. Chem.*, 1895, **51**, 129.
Gabriel, *Ber.*, 1903, **36**, 811.
Fetscher, Bogert, *J. Org. Chem.*, 1939, **4**, 86.

1 : 2 : 3 : 4-Tetrahydroquinoline (Py-*Tetrahydroquinoline*)



$C_9H_{11}N$ MW, 133

B.p. 249–50°/755 mm. $D_4^{23.9}$ 1·0546. Heat of comb. C_v 1226·6 Cal. $n_D^{23.9}$ 1·5933.

B,HCl: prisms from EtOH. M.p. 180–1°.

B,HBr: m.p. 167°.

B,HI: needles from MeOH. M.p. 170°.

N-*Allyl*: b.p. 264–6°/755 mm. *B,HI*: m.p. 141°.

N-*Benzyl*: needles from EtOH. M.p. 36–7°. B.p. 218–22°/38 mm.

N-o-*Nitrobenzyl*: m.p. 111°.

N-m-*Nitrobenzyl*: red prisms from EtOH. M.p. 99°.

N-p-*Nitrobenzyl*: light red cryst. from EtOH. M.p. 102°.

N-*Acetyl*: b.p. 295°.

N-*Benzoyl*: plates from EtOH. M.p. 76°.

N-*Me*: *see* Kairoline.

N-*Et*: *see* Kairoline A.

Hoffmann, Koenigs, *Ber.*, 1883, **16**, 728.
Wedekind, *Ber.*, 1902, **35**, 185.
Lellmann, Pekrun, *Ann.*, 1890, **259**, 50.
Skita, Meyer, *Ber.*, 1912, **45**, 3594.

5 : 6 : 7 : 8-Tetrahydroquinoline (Bz-*Tetrahydroquinoline*).

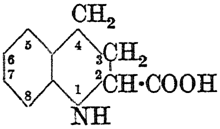
B.p. 222°, 92–5°/12 mm. D_4^{20} 1·0304. n_D^{20} 1·5426. No col. with $FeCl_3$.

B_2,H_2PtCl_6: yellow cryst. Decomp. at 210°.

Picrate: m.p. 157°.

v. Braun, Lemke, *Ann.*, 1930, **478**, 190.

1 : 2 : 3 : 4-Tetrahydroquinoline-2-carboxylic Acid (1 : 2 : 3 : 4-*Tetrahydroquinaldinic acid*)



$C_{10}H_{11}O_2N$ MW, 177

Brownish-yellow cryst. from C_6H_6. M.p. 112–13°.

B,HCl: cryst. + $2H_2O$ from HCl.Aq. M.p. 115–20°, anhyd. 200° decomp.

Me ester: $C_{11}H_{13}O_2N$. MW, 191. B.p. 180°/15 mm. *B,HCl*: cryst. from MeOH–Et_2O. M.p. 191°. *Picrolonate*: yellow prisms. M.p. 172°.

N-*Nitroso*: needles from EtOH, AcOEt or C_6H_6. Decomp. at 132°.

N-*Acetyl*: prisms. M.p. 175–6°.

N-*Benzoyl*: m.p. 187–8°.

Wieland, Hettche, Hoshino, *Ber.*, 1928, **61**, 2377.

1 : 2 : 3 : 4-Tetrahydroquinoline-4-carboxylic Acid (1 : 2 : 3 : 4-*Tetrahydrocinchoninic acid*).

Free acid not isolated.

B,HCl: prisms + H_2O from H_2O. $FeCl_3$ ⟶ brownish-green col. ⟶ green on standing.

Me ester: cryst. from pet. ether. M.p. 88°.

N-*Nitroso*: reddish-yellow cryst. powder. M.p. 69–70°.

N-*Me*: kairoline-4-carboxylic acid. $C_{11}H_{13}O_2N$. MW, 191. Prisms + $2H_2O$ from EtOH. M.p. 169–70° decomp. Very sol. EtOH. Spar. sol. Et_2O, $CHCl_3$, C_6H_6. Bitter taste. *Anhydride*: oil. B.p. 279°/744 mm. Sol. EtOH, Et_2O. Insol. H_2O.

N-*Acetyl*: cryst. from H_2O. M.p. 164·5°. Spar. sol. cold H_2O, EtOH. Insol. Et_2O.

N-*Nitroso*: yellowish needles from H_2O. M.p. 137°. Sol. EtOH, hot H_2O. Less sol. Et_2O.

Weidel, *Monatsh.*, 1882, **3**, 61.
Weidel, Hazura, *Monatsh.*, 1884, **5**, 643.
v. Braun, Lemke, *Ann.*, 1930, **478**, 194.

5 : 6 : 7 : 8-Tetrahydroquinoline-4-carboxylic Acid (5 : 6 : 7 : 8-*Tetrahydrocinchoninic acid*).

Cryst. from H_2O. M.p. 242° decomp. Sol. H_2O. Spar. sol. EtOH. Long heating ⟶ 5 : 6 : 7 : 8-tetrahydroquinoline.

v. Braun, Lemke, *Ann.*, 1930, **478**, 190.

1 : 2 : 3 : 4-Tetrahydroquinoline-5-carboxylic Acid.

Needles or leaflets from EtOH.Aq. M.p. 146–7°.

N-*Me*: kairoline-5-carboxylic acid. Needles from EtOH.Aq. M.p. 164°. Sol. EtOH. Less sol. Et_2O. Spar. sol. H_2O.

N-*Nitroso*: yellow prisms from EtOH.Aq. M.p. 186° decomp.

Fischer, Körner, *Ber.*, 1884, **17**, 765.
Lellmann, Alt, *Ann.*, 1887, **237**, 315.

1 : 2 : 3 : 4-Tetrahydroquinoline-6-carboxylic Acid.

Needles. Decomp. about 170°. Sol. EtOH, Et_2O. C_6H_6. Spar. sol. cold H_2O.

N-*Me*: kairoline-6-carboxylic acid. Cryst. from C_6H_6. M.p. about 224° decomp. Sol. EtOH. Less sol. Et_2O, C_6H_6. Spar. sol. cold H_2O.

N-*Et*: leaflets from C_6H_6. M.p. about 200° decomp.

N-*Acetyl*: needles from H_2O. M.p. 187°. Sol. EtOH, $CHCl_3$, C_6H_6. Less sol. hot H_2O. Insol. Et_2O, ligroin.

N-*Nitroso*: yellowish prisms from C_6H_6. Decomp. about 181°.

Fischer, Endres, *Ber.*, 1902, **35**, 2613.
Kunckell, Vollhase, *Ber.*, 1909, **42**, 3198.

1 : 2 : 3 : 4-Tetrahydroquinoline-7-carboxylic Acid.

Leaflets from EtOH.Aq. M.p. 189°. Sol. MeOH, EtOH, $CHCl_3$. Less sol. Et_2O, C_6H_6. Spar. sol. H_2O.

N-*Me*: kairoline-7-carboxylic acid. Cryst. from C_6H_6. M.p. 185°.

N-*Et*: prisms from C_6H_6. M.p. 163–4°.

N-*Nitroso*: yellowish prisms from C_6H_6. Decomp. at 191°.

Fischer, Endres, *Ber.*, 1902, **35**, 2612.

1 : 2 : 3 : 4-Tetrahydroquinoline-8-carboxylic Acid.

Needles from ligroin or EtOH.Aq. M.p. 163°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, CS_2. Very spar. sol. hot H_2O. Neutral or alkaline sols. show blue fluor. Sublimes.

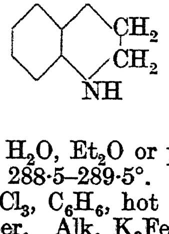
N-*Me*: kairoline-8-carboxylic acid. Needles from C_6H_6. M.p. 218–19°.

N-*Et*: leaflets from EtOH.Aq. M.p. 196–7°.

N-*Nitroso*: plates from C_6H_6. Decomp. about 124°.

Fischer, Endres, *Ber.*, 1902, **35**, 2611.
Tafel, *Ber.*, 1894, **27**, 825.

1 : 2 : 3 : 4-Tetrahydroquinoxaline (*Ethylene-*o*-phenylenediamine*)



$C_8H_{10}N_2$ MW, 134

Leaflets from H_2O, Et_2O or pet. ether. M.p. 96·5–97°. B.p. 288·5–289·5°. Very sol. Et_2O. Sol. EtOH, $CHCl_3$, C_6H_6, hot H_2O. Spar. sol. boiling pet. ether. Alk. $K_3Fe(CN)_6$ ⟶ quinoxaline. $FeCl_3$ ⟶ violet col. with conc. sols., blue with dilute.

N-*Me*: $C_9H_{12}N_2$. MW, 148. Pale yellow oil. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$, pet. ether. Spar. sol. boiling H_2O. $FeCl_3$ ⟶ blue col.

N : N′-*Diacetyl*: cryst. from Et_2O. M.p. 144°. B.p. 350° decomp. Sol. H_2O. Mod. sol. EtOH, $CHCl_3$, C_6H_6. Spar. sol. Et_2O.

N : N′-*Dibenzenesulphonyl*: cryst. from AcOH. M.p. 180°.

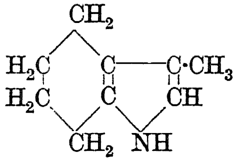
N : N′-*Dinitroso*: pale yellow microneedles from $CHCl_3$–ligroin. M.p. 168° decomp.

I.G., D.R.P., 495,101, (*Chem. Abstracts*, 1930, **24**, 3251).
Hinsberg, Strupler, *Ann.*, 1895, **287**, 225.
Ris, *Ber.*, 1888, **21**, 378.
Merz, Ris, *Ber.*, 1887, **20**, 1191, 1196.
Cavagnol, Wiselogle, *J. Am. Chem. Soc.*, 1947, **69**, 795.

Tetrahydrosantonin.
See Santonan.

Tetrahydrosantoninic Acid.
See Santonanic Acid.

4 : 5 : 6 : 7-Tetrahydroskatole (3-*Methyl-*4 : 5 : 6 : 7-*tetrahydroindole*)

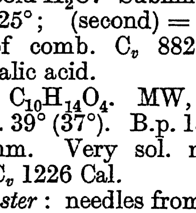


$C_9H_{13}N$ MW, 135

B.p. 105°/12 mm. D_4^{14} 0·9698.

v. Braun, Bayer, Blessing, *Ber.*, 1924, **57**, 400.

Δ[1]-Tetrahydroterephthalic Acid (*Cyclohexene*-1 : 4-*dicarboxylic acid*)



$C_8H_{10}O_4$ MW, 170

Prisms from H_2O. Does not melt below 300°. Sol. 4066 parts cold H_2O. Sublimes. k (first) = $5{\cdot}0 \times 10^{-5}$ at 25°; (second) = $2{\cdot}8 \times 10^{-6}$ at 100°. Heat of comb. C_v 882·8 Cal. Alk. $KMnO_4$ ⟶ oxalic acid.

Di-Me ester: $C_{10}H_{14}O_4$. MW, 198. Needles from H_2O. M.p. 39° (37°). B.p. 153·3–154·5°/20 mm., 147°/9 mm. Very sol. most solvents. Heat of comb. C_v 1226 Cal.

*Di-*l*-menthyl ester*: needles from EtOH. M.p. 125°. $[\alpha]_D^{20}$ − 69·4° in $CHCl_3$.

Diphenyl ester: plates from AcOEt or Me_2CO. M.p. 145°. Sol. EtOH, Et_2O, $CHCl_3$, ligroin.

Rupe, *Ann.*, 1910, **373**, 123.
Baeyer, *Ann.*, 1888, **245**, 159.

Δ²-Tetrahydroterephthalic Acid (*Cyclohexene*-3 : 6-*dicarboxylic acid*).

Cis:

Plates from H_2O. M.p. 150·5°. Sol. 37 parts cold H_2O.

Trans:

Rhombohedra from H_2O. M.p. 220°. Sol. 588 parts cold H_2O. Very sol. hot H_2O.

Di-Me ester: plates or prisms from ligroin. M.p. 3°.

Diphenyl ester: plates from C_6H_6–ligroin. M.p. 107°.

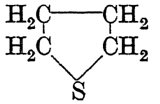
Dibenzyl ester: cryst. from EtOH. M.p. 48°.

Rupe, *Ann.*, 1910, **373**, 123.
Baeyer, *Ann.*, 1889, **251**, 279, 306.
Baeyer, Herb, *Ann.*, 1890, **258**, 39.

Tetrahydrothiazine.
See 1 : 4-Thiazan.

Tetrahydrothiazole.
See Thiazolidine.

Tetrahydrothiophene (*Tetramethylene sulphide*)

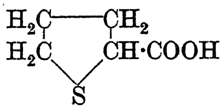


C_4H_8S MW, 88

Mobile liq. with penetrating odour. B.p. 118–19°. D_4^{18} 0·9607. n_D^{18} 1·4871. Volatile in steam. Misc. with most solvents except H_2O. $KMnO_4 \longrightarrow$ sulphone, m.p. 20–1° (8–10°), b.p. 285–8°/743 mm., 153–4°/18 mm., 149·5–150°/15 mm. Forms add. comp. with one mol. $HgCl_2$, needles, m.p. 124·5–125·5°.

Grischkewitsch-Trochimowski, *J. Russ. Phys.-Chem. Soc.*, 1916, **48**, 901.
Bost, Conn, *Chem. Abstracts*, 1933, **27**, 5323.
Backer, Bolt, *Rec. trav. chim.*, 1935, **54**, 539.

Tetrahydrothiophene-2-carboxylic Acid



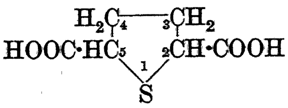
$C_5H_8O_2S$ MW, 132

Plates from H_2O. M.p. 51°. Very sol. H_2O, EtOH. Spar. sol. Et_2O. Heat of comb. C_v 753·3 Cal. $k = 1{\cdot}15 \times 10^{-4}$ at 25°. Volatile in steam. Reduces boiling $NH_3.AgNO_3$.

Me ester: $C_6H_{10}O_2S$. MW, 146. B.p. 206°.

Ernst, *Ber.*, 1887, **20**, 518.

Tetrahydrothiophene-2 : 5-dicarboxylic Acid



$C_6H_8O_4S$ MW, 176

Plates. M.p. 144–5°. Sol. H_2O. Less sol. Et_2O. Reduces warm $NH_3.AgNO_3$.

Di-Et ester: b.p. 157°/10 mm.

Dihydrazide: needles from EtOH. M.p. 208–9°.

Ernst, *Ber.*, 1886, **19**, 3275.
Fredga, *J. prakt. Chem.*, 1938, **150**, 124.
Brown, Kilmer, *J. Am. Chem. Soc.*, 1943 **65**, 1674.

Tetrahydrothiophene-3 : 4-dicarboxylic Acid.

Cis:

White blades from Me_2CO–C_6H_6. M.p. 134–5°.

Anhydride: m.p. 82–5°.

Trans:

Leaflets from C_6H_6. M.p. 131–4°.

Anhydride: m.p. 84–5°.

Dihydrazide: needles from EtOH. M.p. 223–4°.

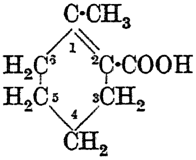
Brown, Baker, Bernstein, Safir, *J. Org. Chem.*, 1947, **12**, 155.

Tetrahydrothiopyran.
See Pentamethylene sulphide.

Tetrahydrothiotolene.
See Methyltetrahydrothiophene.

Tetrahydrotoluene.
See Methylcyclohexene.

Δ¹-Tetrahydro-o-toluic Acid (2-*Methylcyclohexene*-1-*carboxylic acid*)



$C_8H_{12}O_2$ MW, 140

Needles from H_2O. M.p. 87° (79–80°). Sol. EtOH, Me_2CO, pet. ether. Spar. sol. H_2O. Ox. $\longrightarrow$ 3-acetobutyric acid.

Et ester: $C_{10}H_{16}O_2$. MW, 168. B.p. 148°/100 mm.

Kay, Perkin, *J. Chem. Soc.*, 1905, **87**, 1068.
Mazza, di Mase, *Gazz. chim. ital.*, 1927, **57**, 300.

Δ²-Tetrahydro-o-toluic Acid (6-*Methylcyclohexene*-1-*carboxylic acid*).

Cryst. from pet. ether. M.p. 80°.

Mazza, Cremona, *Gazz. chim. ital.*, 1927, **57**, 318.

Δ³-Tetrahydro-o-toluic Acid (4-*Methylcyclohexene*-3-*carboxylic acid*).

Cis:

B.p. 145–6°/20 mm. Ox. $\longrightarrow$ *trans*-form.

Et ester: b.p. 143–4°/100 mm.

Trans:
M.p. 60–2°. B.p. 162–3°/50 mm.
Et ester: b.p. 144–6°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 744.

Δ^4-Tetrahydro-o-toluic Acid (5-*Methyl-cyclohexene*-4-*carboxylic acid*).
Thick syrup. B.p. 180–1°/100 mm., 143°/20 mm., 135°/12 mm. Ox. ⟶ methylbutane-1 : 2 : 4-tricarboxylic acid.
Et ester: b.p. 140°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 754.

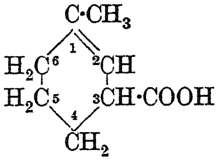
Δ^5-Tetrahydro-o-toluic Acid (3-*Methyl-cyclohexene*-4-*carboxylic acid*).
B.p. 139°/20 mm., 114–30°/9 mm.
Et ester: b.p. 204–8°, 138–9°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 735.
Skita, Ardan, Krauss, *Ber.*, 1908, **41**, 2944.

Δ^6-Tetrahydro-o-toluic Acid (2-*Methyl-cyclohexene*-3-*carboxylic acid*).
B.p. 140–2°/20 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 738.

Δ^1-Tetrahydro-m-toluic Acid (1-*Methyl-cyclohexene*-3-*carboxylic acid*)



$C_8H_{12}O_2$ MW, 140

l-.
$[\alpha]_D$ − 49·7° in AcOEt.
Me ester: $C_9H_{14}O_2$. MW, 154. B.p. 144–7°/100 mm.

dl-.
B.p. 140–2°/20 mm., 123°/7 mm. Ox. ⟶ adipic acid + 4-aceto-*n*-valeric acid.
Et ester: $C_{10}H_{16}O_2$. MW, 168. B.p. 128°/60 mm.

Perkin, Tattersall, *J. Chem. Soc.*, 1907, **91**, 496.
Haworth, Perkin, *J. Chem. Soc.*, 1913, **103**, 2237.
Boorman, Linstead, *J. Chem. Soc.*, 1935, 264.

Δ^2-Tetrahydro-m-toluic Acid (3-*Methyl-cyclohexene*-1-*carboxylic acid*).
M.p. 26°. B.p. 150°/11 mm., 130°/7 mm. Spar. sol. H_2O. Ox. ⟶ 1-methyladipic acid.

Boorman, Linstead, *J. Chem. Soc.*, 1935, 264.
Perkin, Tattersall, *J. Chem. Soc.*, 1905, **87**, 1095.

Δ^3-Tetrahydro-m-toluic Acid (5-*Methyl-cyclohexene*-1-*carboxylic acid*).

d-.
M.p. 62–4°. $[\alpha]_D$ + 40·1° in AcOEt. Volatile in steam.
Et ester: b.p. 150–1°/100 mm. $[\alpha]_D$ + 32·5° in AcOEt.

l-.
$[\alpha]_D$ − 35·8° in AcOEt.

dl-.
Plates from formic acid. M.p. 60°. B.p. 155–60°/25 mm. Very sol. most solvents. Ox. ⟶ 2-methyladipic acid.
Et ester: b.p. 146–8°/100 mm., 105°/11 mm. D_4^{18} 0·9762. n_{He}^{20} 1·4695.

Auwers, *Ann.*, 1923, **432**, 98.
Perkin, Tattersall, *J. Chem. Soc.*, 1905, **87**, 1093.
Luff, Perkin, *J. Chem. Soc.*, 1910, **97**, 2151.

Δ^4-Tetrahydro-m-toluic Acid (5-*Methyl-cyclohexene*-3-*carboxylic acid*).
B.p. 143–6°/20 mm.
Et ester: b.p. 142–4°/100 mm.

Perkin, *J. Chem. Soc.*, 1910, **97**, 2146.

Δ^5-Tetrahydro-m-toluic Acid (6-*Methyl-cyclohexene*-4-*carboxylic acid*).

d-.
B.p. 142–5°/20 mm. $[\alpha]_D^{16}$ + 33° in AcOEt.
Et ester: b.p. 140–1°/100 mm. $[\alpha]_D^{16}$ + 30·5° in AcOEt.

l-.
B.p. 142°/20 mm. $[\alpha]_D^{15}$ − 30·9° in AcOEt.
Et ester: b.p. 140–2°/100 mm. $[\alpha]_D^{15}$ − 27·4° in AcOEt.

dl-.
B.p. 177–80°/100 mm., 145°/20 mm.
Et ester: b.p. 141–3°/100 mm.

Perkin, *J. Chem. Soc.*, 1910, **97**, 2139.

Δ^6-Tetrahydro-m-toluic Acid (2-*Methyl-cyclohexene*-4-*carboxylic acid*).

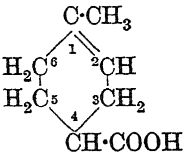
d-.
B.p. 138–40°/18 mm. $[\alpha]_D$ + 108° in AcOEt.

l-.
$[\alpha]_D$ − 98·6° in AcOEt.

dl-.
B.p. 184–6°/100 mm.
Et ester: b.p. 146°/100 mm.

Fisher, Perkin, *J. Chem. Soc.*, 1908, **93**, 1886.
Haworth, Perkin, *J. Chem. Soc.*, 1913, **103**, 2233.

Δ¹-Tetrahydro-*p*-toluic Acid (1-*Methylcyclohexene*-4-*carboxylic acid*)



$C_8H_{12}O_2$ MW, 140

d-.

M.p. about 99°. $[\alpha]_D + 55°$.

Et ester: $C_{10}H_{16}O_2$. MW, 168. B.p. 145–7°/100 mm. $[\alpha]_D + 50°$.

l-.

Cryst. from pet. ether. M.p. about 99°. $[\alpha]_D - 58°$.

Et ester: b.p. 145–7°/100 mm. $[\alpha]_D - 52°$.

dl-.

Needles from H_2O. M.p. 99°. B.p. 140–5°/15 mm. Sol. AcOH. Spar. sol. H_2O. Conc. $H_2SO_4 \longrightarrow$ *p*-toluic acid.

Et ester: b.p. 155–7°/100 mm., 105–8°/12 mm.

Chloride: $C_8H_{11}OCl$. MW, 158·5. B.p. 110–14°/40 mm., 90°/19 mm.

Amide: $C_8H_{13}ON$. MW, 139. Needles or leaflets from H_2O. M.p. 182°.

Lehmann, Paasche, *Ber.*, 1935, **68**, 1069.
Meldrum, Perkin, *J. Chem. Soc.*, 1908, **93**, 1424.

Δ³-Tetrahydro-*p*-toluic Acid (4-*Methylcyclohexene*-1-*carboxylic acid*).

d-.

Prisms from H_2O. M.p. 136–7° (133°). $[\alpha]_D + 100·1°$ in AcOEt.

Et ester: b.p. 154°/100 mm. $[\alpha]_D + 86·5°$. D_{20}^{20} 0·9757. n_D^{20} 1·4688.

l-.

Prisms from AcOEt. M.p. 133–4°. Sol. AcOH, AcOEt, pet. ether. Spar. sol. cold EtOH. $[\alpha]_D^{17} - 100·8°$ in AcOEt.

Et ester: b.p. 154°/100 mm. $[\alpha]_D^{18} - 83·5°$.

dl-.

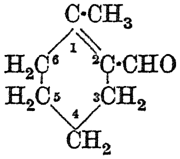
Prismatic needles from AcOH.Aq. M.p. 132–4°. Very sol. EtOH, Et_2O, AcOH, $CHCl_3$, C_6H_6. Less sol. cold CS_2, ligroin.

Et ester: b.p. 152–3°/100 mm. D_{15}^{15} 0·9792. n_α^{15} 1·4659.

Amide: leaflets from EtOH.Aq. M.p. 148°. Sol. EtOH. Spar. sol. H_2O.

Einhorn, Willstätter, *Ann.*, 1894, **280**, 163.
Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 645.
Kay, Perkin, *J. Chem. Soc.*, 1906, **89**, 844.
Chou, Perkin, *J. Chem. Soc.*, 1911, **99**, 534.

Δ¹-Tetrahydro-*o*-toluic Aldehyde (2-*Methylcyclohexene*-1-*aldehyde*)



$C_8H_{12}O$ MW, 124

Needles. M.p. 98–101°. B.p. 75–6°/12 mm.

Semicarbazone: cryst. from MeOH. M.p. 208–12°. Insol. H_2O.

p-*Nitrophenylhydrazone*: cryst. from AcOH. M.p. 160–3°.

Bernhauer, Neubauer, *Biochem. Z.*, 1932, **251**, 173.

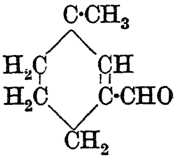
Δ⁴-Tetrahydro-*o*-toluic Aldehyde (5-*Methylcyclohexene*-4-*aldehyde*).

Liq. B.p. 83°/38 mm., 75°/22 mm.

Semicarbazone: m.p. 168°.

Diels, Alder, *Ann.*, 1929, **470**, 85.
I.G., U.S.P., 1,944,731, (*Chem. Abstracts*, 1934, **28**, 2016).

Δ²-Tetrahydro-*m*-toluic Aldehyde (3-*Methylcyclohexene*-1-*aldehyde*)

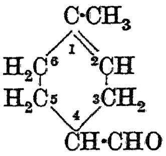


$C_8H_{12}O$ MW, 124

Semicarbazone: m.p. 206–7°. Spar. sol. most solvents.

Wallach, *Ann.*, 1906, **347**, 344.

Δ¹-Tetrahydro-*p*-toluic Aldehyde (1-*Methylcyclohexene*-4-*aldehyde*)



$C_8H_{12}O$ MW, 124

B.p. 63–4°/10 mm.

Semicarbazone: m.p. 146°.

Diels, Alder, *Ann.*, 1929, **470**, 87.
See also Lehmann, Paasche, *Ber.*, 1935, **68**, 1068.

Δ³-Tetrahydro-*p*-toluic Aldehyde (4-*Methylcyclohexene*-1-*aldehyde*).

Semicarbazone: m.p. 192–4°.

Wallach, Evans, *Ann.*, 1906, **347**, 346.

Tetrahydrotoluquinoline.

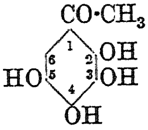
See 6-, 7-, and 8-Methyltetrahydroquinolines.

Tetrahydrotolylacetic Acid.

See Methylcyclohexenylacetic Acid.

Tetrahydrotubanol.
See 2-Isoamylresorcinol.

2 : 3 : 4 : 5-Tetrahydroxyacetophenone



$C_8H_8O_5$ MW, 184

3 : 4-*Di-Me ether*: $C_{10}H_{12}O_5$. MW, 212. Yellow needles from Et_2O. M.p. 119–21°. Sol. EtOH, $CHCl_3$, C_6H_6. Conc. H_2SO_4 ⟶ yellow sol.

Bargellini, *Gazz. chim. ital.*, 1916, **46**, i, 253.

2 : 3 : 4 : 6-Tetrahydroxyacetophenone.
Pale yellow needles from EtOH or AcOH.Aq. M.p. 204–5°.
3 : 4-*Di-Me ether*: needles from EtOH.Aq. M.p. 166–8° (162–3°). Conc. H_2SO_4 ⟶ orange-yellow sol.
2 : 3 : 4-*Tri-Me ether*: $C_{11}H_{14}O_5$. MW, 226. Needles from EtOH. M.p. 164–5°.
3 : 4 : 6-*Tri-Me ether*: cubes from EtOH. M.p. 112–13° (125–6°). *Acetyl*: m.p. 106°. *Benzoyl*: m.p. 120–2°.
Tetra-Me ether: $C_{12}H_{16}O_5$. MW, 240. Needles from EtOH.Aq. M.p. 53–4° (43–5°). B.p. 310°. *Oxime*: m.p. 103–5°. *Semicarbazone*: m.p. 128–30°.
Phenylhydrazone: red prismatic needles. M.p. 248–51° decomp.

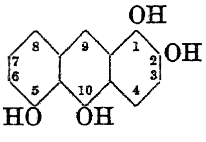
Bargellini, Bini, *Gazz. chim. ital.*, 1911, **41**, ii, 18.
Bargellini, Zoras, *Gazz. chim. ital.*, 1934, **64**, 192.
Bargellini, *Gazz. chim. ital.*, 1919, **49**, ii, 47.
Nierenstein, *J. Chem. Soc.*, 1917, **111**, 6.

Tetrahydroxyacetophenone.
See also Trihydroxyphenacyl Alcohol.

Tetrahydroxyadipic Acid.
See Mucic Acid, Allomucic Acid *and* Saccharic Acid.

Tetrahydroxy-1-aminocaproic Acid.
See Glucosaminic Acid.

1 : 2 : 5 : 10-Tetrahydroxyanthracene
(1 : 5 : 6-*Trihydroxyanthrone*, 1 : 5 : 6-*trihydroxyanthranol*)



$C_{14}H_{10}O_4$ MW, 242

Cryst. from EtOH.Aq. M.p. 258°.
Mono-Me ether: $C_{15}H_{12}O_4$. MW, 256. Cryst. from EtOH. M.p. 140°.

Graebe, Thode, *Ann.*, 1906, **349**, 218.

1 : 2 : 6 : 10-Tetrahydroxyanthracene
(2 : 5 : 6-*Trihydroxyanthrone*, 2 : 5 : 6-*trihydroxyanthranol*.
Yellow cryst. M.p. 258°. NaOH ⟶ reddish-yellow sol. Conc. H_2SO_4 ⟶ brownish-yellow sol.
Tri-Me ether: $C_{17}H_{16}O_4$. MW, 284. Needles from C_6H_6. M.p. 169–70°. Sol. hot C_6H_6. Mod. sol. hot EtOH, AcOH.
Tetra-acetyl: m.p. 250–60°.

Bayer, D.R.P., 117,923, (*Chem. Zentr.*, 1901, I, 600).
Graebe, Thode, *Ann.*, 1906, **349**, 214.
Bistrzycki, Yssel de Schepper, *Ber.*, 1898, **31**, 2799.

1 : 2 : 7 : 10-Tetrahydroxyanthracene
(3 : 4 : 6-*Trihydroxyanthrone*, 3 : 4 : 6-*trihydroxyanthranol*).
Yellow needles.
1 : 2 : 7-*Tri-Me ether*: needles from EtOH. M.p. 149°. *Acetyl*: needles. M.p. 127°.
Tetra-acetyl: needles from EtOH. M.p. 167°.

MacMaster, Perkin, *J. Chem. Soc.*, 1927, 1309.
Graebe, Bernhard, *Ann.*, 1906, **349**, 227.
Liebermann, *Ber.*, 1888, **21**, 443.

1 : 2 : 9 : 10-Tetrahydroxyanthracene
(1 : 2 : 10-*Trihydroxyanthrone*, 1 : 2 : 10-*trihydroxyanthranol*, 1 : 2-*dihydroxyanthrahydroquinone*, *leuco-alizarin*).
Brown leaflets from AcOH. M.p. 150°. Very sol. EtOH with yellow col. Conc. H_2SO_4 ⟶ brown col. ⟶ red on standing. NaOH ⟶ red sol.

Grandmougin, *J. prakt. Chem.*, 1907, **76**, 141.

1 : 3 : 9 : 10-Tetrahydroxyanthracene
(*Dihydropurpuroxanthin*, *leuco-purpuroxanthin*, 1 : 3 : 10-*trihydroxyanthrone*, 1 : 3 : 10-*trihydroxyanthranol*, 1 : 3-*dihydroxyanthrahydroquinone*).
Pale yellow needles. Sol. Et_2O. NaOH ⟶ brown sol. Oxidises in air to purpuroxanthin.

Rosenstiehl, *Ann. chim. phys.*, 1879, **18**, 230.

1 : 4 : 9 : 10-Tetrahydroxyanthracene (*Dihydroquinizarin*, *leuco-quinizarin*, 1 : 4 : 10-*trihydroxyanthrone*, 1 : 4 : 10-*trihydroxyanthranol*, 1 : 4-*dihydroxyanthrahydroquinone*).
Yellow needles. M.p. 156° (131–6°). Alkalis ⟶ red sols. Sol. EtOH, AcOH with yellow col. and weak blue fluor. Conc. H_2SO_4 ⟶ yellow sol.
Tetra-acetyl: pale yellow cryst. from AcOH. M.p. 240–2°.

M.L.B., D.R.P., 207,668, (*Chem. Zentr.*, 1909, I, 1287).
Zahn, Ochwat, *Ann.*, 1928, **462**, 72.
Meyer, Sander, *Ann.*, 1920, **420**, 123.

2 : 3 : 6 : 7-Tetrahydroxyanthracene.
Tetra-Me ether: $C_{18}H_{18}O_4$. MW, 298. Cryst. from EtOH. M.p. 173°. Most sols. show blue fluor.

Robinson, *J. Chem. Soc.*, 1915, **107**, 272.

2 : 3 : 9 : 10-Tetrahydroxyanthracene (*Dihydrohystazarin*, 2 : 3 : 9-*trihydroxyanthrone*, 2 : 3 : 9-*trihydroxyanthranol*, *leuco-hystazarin*).
Tetra-acetyl: needles from EtOH. M.p. 217–19°.

Schöller, *Ber.*, 1889, **22**, 684.

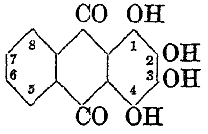
2 : 6 : 9 : 10-Tetrahydroxyanthracene (*Dihydroanthraflavic acid*, 2 : 6 : 10-*trihydroxyanthrone*, 2 : 6 : 10-*trihydroxyanthranol*, *leucoanthraflavic acid*).
Tetra-acetyl: needles. M.p. 274°.

Liebermann, *Ber.*, 1888, **21**, 1173.

2 : 7 : 9 : 10-Tetrahydroxyanthracene (*Dihydroisoanthraflavic acid*, 2 : 7 : 10-*trihydroxyanthrone*, 2 : 7 : 10-*trihydroxyanthranol*, *leuco-isoanthraflavic acid*).
Tetra-acetyl: needles. M.p. 235–40°.

Liebermann, *Ber.*, 1888, **21**, 1173.

1 : 2 : 3 : 4-Tetrahydroxyanthraquinone



$C_{14}H_8O_6$ MW, 272

Green needles. Conc. $H_2SO_4 \longrightarrow$ red sol. KOH $\longrightarrow$ red sol.
Tetra-acetyl: yellow needles. M.p. 205°. Sol. boiling KOH with violet-red col.

Slama, *Chem. Zentr.*, 1899, II, 966.
Bayer, D.R.P., 125,579, (*Chem. Zentr.*, 1901, II, 1188).

1 : 2 : 3 : 5-(or 1 : 2 : 3 : 7-) Tetrahydroxyanthraquinone (*α-Hydroxyanthragallol*).
Yellow needles from EtOH. Does not melt below 350°. Conc. $H_2SO_4 \longrightarrow$ violet sol.
Tetra-acetyl: needles from AcOH. M.p. 207–9°.

Liebermann, Kostanecki, *Ann.*, 1888, **244**, 360; 1887, **240**, 270.
Briggs *et al.*, *J. Chem. Soc.*, 1948, 990.
Wiles, *J. Chem. Soc.*, 1952, 1358.

1 : 2 : 3 : 7-(or 1 : 2 : 3 : 5-) Tetrahydroxyanthraquinone (*β-Hydroxyanthragallol*).
Red needles from EtOH. M.p. 380°. Conc. $H_2SO_4 \longrightarrow$ brownish-yellow sol.
Tetra-acetyl: yellow plates from AcOH. M.p. 189°. Sol. EtOH, $CHCl_3$, AcOH.

Liebermann, Kostanecki, Noah, *Ann.*, 1888, **244**, 360; 1887, **240**, 271.
Briggs *et al.*, *J. Chem. Soc.*, 1948, 990.
Wiles, *J. Chem. Soc.*, 1952, 1358.

1 : 2 : 4 : 6 - Tetrahydroxyanthraquinone (*Hydroxyflavopurpurin*).
Cryst. Sol. EtOH, Py. Less sol. AcOH. Conc. $H_2SO_4 \longrightarrow$ bluish-red sol.
Tetra-acetyl: m.p. 202°.

Dimroth, Fick, *Ann.*, 1916, **411**, 324.
Bayer, D.R.P., 67,061.

1 : 2 : 4 : 7 - Tetrahydroxyanthraquinone (*Hydroxyanthrapurpurin*).
Cryst. from EtOH, Py, or AcOH. Sol. EtOH, AcOH. NaOH $\longrightarrow$ bluish-red sol. Conc. $H_2SO_4 \longrightarrow$ cherry-red sol.
2 : 7-*Diacetyl*: orange needles from AcOH. M.p. 224–5°. Sol. AcOEt.
Tetra-acetyl: m.p. 214·5°.

Dimroth, Friedemann, Kammerer, *Ber.*, 1920, **53**, 483.
Dimroth, Fick, *Ann.*, 1916, **411**, 328.
Bayer, D.R.P., 67,061.

1 : 2 : 5 : 6-Tetrahydroxyanthraquinone. *See* Rufiopin.

1 : 2 : 5 : 8-Tetrahydroxyanthraquinone. *See* Quinalizarin.

1 : 2 : 6 : 7-Tetrahydroxyanthraquinone.
Orange needles from $PhNO_2$. Does not melt below 330°. Mod. sol. $PhNO_2$. Spar. sol. EtOH, C_6H_6. Conc. $H_2SO_4 \longrightarrow$ violet sol. NaOH $\longrightarrow$ violet sol.
1 : 2 : 6-*Tri-Me ether*: $C_{17}H_{14}O_6$. MW, 314. Yellow needles from AcOH. M.p. 269–70°.
Tetra-Me ether: $C_{18}H_{16}O_6$. MW, 328. Yellow needles from AcOH. M.p. 244–5° (239°).
Tetra-acetyl: cryst. from AcOH. M.p. 239–41°.

Bistrzycki, Krauer, *Helv. Chim. Acta*, 1923, **6**, 768.
Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2017.

1 : 2 : 7 : 8-Tetrahydroxyanthraquinone.
Red prisms from AcOH. M.p. 315° (292°) decomp. Sol. Py. Spar. sol. EtOH, AcOH, $PhNO_2$, toluene. NaOH $\longrightarrow$ blue sol. Conc. $H_2SO_4 \longrightarrow$ violet-red sol. Sublimes.
Diacetyl deriv.: m.p. 242°.
Tetra-acetyl: yellow needles. M.p. 238–40° decomp.

Bayer, D.R.P., 103,988, (*Chem. Zentr.*, 1899, II, 922).
Heller, *Z. angew. Chem.*, 1929, **42**, 170.

1 : 3 : 5 : 7-Tetrahydroxyanthraquinone. *See* Anthrachrysazin.

1 : 4 : 5 : 8-Tetrahydroxyanthraquinone.
Brown needles from C_6H_6–ligroin. Does not melt below 300° (246°). Insol. H_2O. NaOH $\longrightarrow$ blue sol. Conc. $H_2SO_4 \longrightarrow$ greenish-blue sol. with red. fluor.
Tetra-Me ether: orange leaflets from AcOH. M.p. 317°. Sol. boiling AcOH, xylene, $PhNO_2$. Spar. sol. EtOH, C_6H_6, hot H_2O. Conc. H_2SO_4 $\longrightarrow$ greenish-blue sol.

Tetra-acetyl: pale yellow needles from AcOH. M.p. 281–2°. Conc. H_2SO_4 ⟶ blue col.

Fischer, Ziegler, *J. prakt. Chem.*, 1912, **86**, 300.
Frey, *Ber.*, 1912, **45**, 1361.
Bayer, D.R.P., 143,804, (*Chem. Zentr.*, 1903, II, 475).
S.C.I., Bâle, D.R.P., 533,340, (*Chem. Abstracts*, 1932, **26**, 480).
Marshall, *J. Chem. Soc.*, 1937, 255.

1 : 4 : 6 : 7-Tetrahydroxyanthraquinone.
Tetra-acetyl: cryst. from MeOH. M.p. 192–3°.

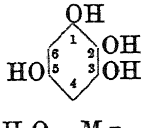
Allen, Frame, Wilson, *J. Org. Chem.*, 1942, **7**, 63.

2 : 3 : 6 : 7-Tetrahydroxyanthraquinone. Orange-yellow. Turns brown at 360°. Sol. alkalis with greenish-yellow col.
Tetra-Me ether: yellow needles from AcOH or $PhNO_2$. M.p. 346° (344°). Conc. H_2SO_4 ⟶ green sol. Sublimes.
2 : 6-*Di-Me*-3 : 7-*di-Et ether*: $C_{20}H_{20}O_6$. MW, 356. Yellow needles from AcOH. M.p. 288°. Conc. H_2SO_4 ⟶ green sol.
2 : 3 : 6-*Tri-Me*-7-*Et ether*: $C_{19}H_{18}O_6$. MW, 342. Yellow needles from AcOH. M.p. 290°. Conc. H_2SO_4 ⟶ green sol.
Tetra-acetyl: yellow cryst. from AcOH. M.p. 300° decomp.

Vanzetti, Dreyfus, *Gazz. chim. ital.*, 1934, **64**, 392.
Vanzetti, Oliverio, *Gazz. chim. ital.*, 1930, **60**, 620.
Haworth, Mavin, *J. Chem. Soc.*, 1931, 1365.

1 : 2 : 3 : 4-Tetrahydroxybenzene.
See Apionol.

1 : 2 : 3 : 5-Tetrahydroxybenzene



$C_6H_6O_4$ MW, 142

Needles from H_2O. M.p. 165°. Sol. H_2O, EtOH, AcOEt. Insol. $CHCl_3$, C_6H_6. Decomp. easily in air. $FeCl_3$ ⟶ red col.
2-*Me ether*: *see* Iretol.
1 : 3-*Di-Me ether*: $C_8H_{10}O_4$. MW, 170. Needles. M.p. 159°. Sol. EtOH, Et_2O. Sol. alkalis with green col. Decomp. in moist air. 2-*Et ether*: $C_{10}H_{14}O_4$. MW, 198. Yellowish needles from ligroin. M.p. 119°. Sol. most org. solvents. $FeCl_3$ ⟶ green col. *Diacetyl*: needles from boiling H_2O. M.p. 128°. Sol. EtOH. Spar. sol. H_2O.
1 : 2 : 3-*Tri-Me ether*: *see* Antiarol.
Tetra-Me ether: *see under* Antiarol.
2-*Et ether*: $C_8H_{10}O_4$. MW, 170. Needles. M.p. 220°. *Triacetyl*: cryst. from C_6H_6–ligroin. M.p. 74°. B.p. 232°/17 mm.

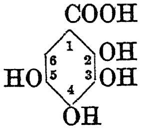
1 : 2 : 3-*Tri-Et ether*: $C_{12}H_{18}O_4$. MW, 226. Needles from H_2O. M.p. 105°. Sol. EtOH, Et_2O.

Oettinger, *Monatsh.*, 1895, **16**, 256.
Will, *Ber.*, 1888, **21**, 610.
Kohner, *Monatsh.*, 1899, **20**, 938.
Nierenstein, *J. Chem. Soc.*, 1917, **111**, 5.

1 : 2 : 4 : 5-Tetrahydroxybenzene. Leaflets from AcOH. M.p. 215–20°. Very sol. H_2O, EtOH, Et_2O. Less sol. AcOH. Aq. sol. turns brown in air.
2-*Me ether*: *triacetyl*, cryst. from MeOH or AcOH. M.p. 142°.
1 : 4-*Di-Me ether*: needles. M.p. 170° (166°). Sol. EtOH, warm $CHCl_3$.
Tetra-Me ether: $C_{10}H_{14}O_4$. MW, 198. Needles from H_2O. M.p. 103°. Sol. EtOH, C_6H_6. Spar. sol. cold H_2O.
1 : 4-*Di-Et ether*: $C_{10}H_{14}O_4$. MW, 198. Needles from hot H_2O. M.p. 138°. *Diacetyl*: leaflets. M.p. 148°.
Tetra-Et ether: $C_{14}H_{22}O_4$. MW, 254. Leaflets from EtOH.Aq. M.p. 143°. Sublimes. Volatile in steam.
1 : 4-*Dipropyl ether*: $C_{12}H_{18}O_4$. MW, 226. Needles from ligroin. M.p. 95°. Very sol. most solvents. Conc. H_2SO_4 ⟶ greenish sol.
Tetra-acetyl: leaflets from AcOH. M.p. 226–7° (217°).

Jackson, Beggs, *J. Am. Chem. Soc.*, 1914, **36**, 1216.
Nietzki, Schmidt, *Ber.*, 1888, **21**, 2377.
Schüler, *Arch. Pharm.*, 1907, **245**, 281.
Nietzki, Rechberg, *Ber.*, 1890, **23**, 1214.
Erdtmann, *Chem. Abstracts*, 1934, **28**, 1337.

2 : 3 : 4 : 5-Tetrahydroxybenzoic Acid



$C_7H_6O_6$ MW, 186

2 : 5-*Di-Me ether*: $C_9H_{10}O_6$. MW, 214. Cryst. from AcOEt. M.p. 147–8°. Sol. H_2O, EtOH, Et_2O, AcOEt. Insol. ligroin. $FeCl_3$ ⟶ blue col. *Di-Et ether*: $C_{13}H_{18}O_6$. MW, 270. Pale yellow cryst. from pet. ether. M.p. 83°. B.p. 95°/0·2 mm.
3 : 5-*Di-Me ether*: pale yellow cryst. M.p. 165° decomp. Sol. EtOH, C_6H_6. $FeCl_3$ ⟶ brown col. *Diacetyl*: cryst. Decomp. at 112° (162°). Sol. EtOH. Insol. H_2O.
3 : 4 : 5-*Tri-Me ether*: $C_{10}H_{12}O_6$. MW, 228. Cryst. M.p. 191° decomp. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O, ligroin. *Me ester*: $C_{11}H_{14}O_6$. MW, 242. Cryst. M.p. 85°. Sol. EtOH, Et_2O. Insol. H_2O.
Tetra-Me ether: $C_{11}H_{14}O_6$. MW, 242.

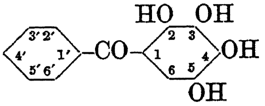
Needles from EtOH.Aq. M.p. 87·5°. Sol. EtOH, boiling H_2O, Me_2CO, AcOEt, C_6H_6.

Bartolotti, *Gazz. chim. ital.*, 1892, **22**, i, 562.
Hamburg, *Monatsh.*, 1898, **19**, 603.
Wesseley, Demmer, *Ber.*, 1928, **61**, 1281.
Bogert, Plant, *J. Am. Chem. Soc.*, 1915, **37**, 2733.

2 : 3 : 4 : 6-Tetrahydroxybenzoic Acid.
Needles from H_2O. M.p. 308–10° decomp.
Tetra-Me ether: needles from EtOH. M.p. 184–6° (149–50°). *Me ester*: $C_{12}H_{16}O_6$. MW, 256. Needles from C_6H_6. M.p. 134–6°. *Chloride*: $C_{11}H_{13}O_5Cl$. MW, 260·5. Needles from pet. ether. M.p. 104°. *Nitrile*: $C_{11}H_{13}O_4N$. MW, 223. Needles from EtOH. M.p. 114°.
Tetra-acetyl: needles from MeOH. M.p. 274–6°.
Tetrabenzoyl: prismatic needles from EtOH. M.p. 248–9°.

Nierenstein, *J. Chem. Soc.*, 1917, **111**, 5.
Bargellini, Madesani, *Gazz. chim. ital.*, 1931, **61**, 684.

2 : 3 : 4 : 5-Tetrahydroxybenzophenone



$C_{13}H_{10}O_5$ MW, 246

3 : 4-*Di-Me ether*: $C_{15}H_{14}O_5$. MW, 274. Yellow prisms from H_2O. M.p. 140–2°. Sol. EtOH, AcOH, C_6H_6. Conc. H_2SO_4 ⟶ yellow sol.

Bargellini, *Gazz. chim. ital.*, 1916, **46**, i, 254.

2 : 3 : 4 : 6-Tetrahydroxybenzophenone.
Tri-Me ether: $C_{16}H_{16}O_5$. MW, 288. Needles from ligroin. M.p. 87–9°. Sol. EtOH, $CHCl_3$, Me_2CO, C_6H_6. Insol. H_2O. Alc. $FeCl_3$ ⟶ reddish-brown col. Conc. H_2SO_4 ⟶ yellow sol. ⟶ green ⟶ dark violet on warming. *Acetyl*: needles from EtOH.Aq. M.p. 130–2°. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6.
Tetra-Me ether: $C_{17}H_{18}O_5$. MW, 302. Needles from ligroin. M.p. 125–6°. Sol. EtOH, Me_2CO, C_6H_6. Insol. H_2O. Conc. H_2SO_4 ⟶ yellow sol. ⟶ dark violet on warming.

Bargellini, *Gazz. chim. ital.*, 1915, **45**, i, 89.

2 : 3 : 4 : 2′-Tetrahydroxybenzophenone.
Yellow plates + H_2O from H_2O. M.p. 100°, anhyd. 149°. Sol. EtOH, AcOH, Et_2O. Spar. sol. C_6H_6, ligroin. Sublimes.
Tetra-acetyl: cryst. from EtOH or AcOH. M.p. 118°.

Badische, D.R.P., 49,149.
Graebe, Eichengrun, *Ann.*, 1892, **269**, 307.
Atkinson, Heilbron, *J. Chem. Soc.*, 1926, 2690.

2 : 3 : 4 : 3′-Tetrahydroxybenzophenone.
Yellow needles. M.p. 133°.

Badische, D.R.P., 49,149.

2 : 3 : 4 : 4′-Tetrahydroxybenzophenone.
Yellowish needles. M.p. above 200°.

Badische, D.R.P., 49,149.

2 : 4 : 5 : 4′-Tetrahydroxybenzophenone.
4 : 5 : 4′-*Tri-Me ether*: yellow needles from EtOH.Aq. M.p. 127–8°. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6. Alc. $FeCl_3$ ⟶ yellowish-green col.
Tetra-Me ether: yellow cryst. powder. M.p. 122–4°. Sol. Me_2CO, $CHCl_3$, C_6H_6. Conc. H_2SO_4 ⟶ orange-yellow sol. *Phenylhydrazone*: m.p. 173–4°.

Bargellini, Martegiani, *Atti accad. Lincei*, 1911, **20**, II, 188.

2 : 4 : 6 : 2′-Tetrahydroxybenzophenone.
Golden-yellow leaflets from EtOH. Blackens on heating. Very spar. sol. EtOH. Insol. H_2O, most org. solvents. Has paralytic action on heart in large doses.

Karrer, *Helv. Chim. Acta*, 1921, **4**, 992.

2 : 4 : 6 : 3′-Tetrahydroxybenzophenone.
Pale yellow leaflets from H_2O. Decomp. at 246°. Sol. EtOH, hot H_2O. Spar. sol. Et_2O. Alc. $FeCl_3$ ⟶ purple col. Alkalis ⟶ orange sols. Conc. H_2SO_4 ⟶ yellow sol.

Nishikawa, Robinson, *J. Chem. Soc.*, 1922, **121**, 842.

2 : 4 : 6 : 4′-Tetrahydroxybenzophenone.
Pale brown prisms from H_2O. M.p. 210°. Alc. $FeCl_3$ ⟶ brown col. Alkalis ⟶ yellow sols.
Tetra-Me ether: prisms from EtOH. M.p. 146°.
4′-*Carbethoxyl*: yellow needles from EtOH.Aq. M.p. 172°.

Nishikawa, Robinson, *J. Chem. Soc.*, 1922, **121**, 843.
Kostanecki, Tambor, *Ber.*, 1906, **39**, 4024.

2 : 3 : 2′ : 3′-Tetrahydroxybenzophenone.
Tetra-Me ether: cryst. M.p. 145°.

Staudinger, Schlenker, Goldstein, *Helv. Chim. Acta*, 1921, **4**, 341.

2 : 4 : 2′ : 4′-Tetrahydroxybenzophenone.
Yellow needles + $1\frac{1}{2}H_2O$ from H_2O. M.p. 193–5°. Sol. MeOH, EtOH, Et_2O, Me_2CO, AcOH. Mod. sol. hot H_2O, $CHCl_3$, C_6H_6, $FeCl_3$ ⟶ dark brown col.
Tetra-Me ether: yellowish leaflets from EtOH.Aq. M.p. 130°. Conc. H_2SO_4 ⟶ yellow sol.

Tambor, *Ber.*, 1910, **43**, 1889.
Meyer, Conzetti, *Ber.*, 1897, **30**, 971.
Shoesmith, Haldane, *J. Chem. Soc.*, 1924, **125**, 113.

2 : 4 : 2′ : 6′-Tetrahydroxybenzophenone. Cryst. + H_2O from H_2O. M.p. about 200°. Sol. hot H_2O.

Graebe, *Ann.*, 1889, **254**, 302.

2 : 4 : 3′ : 4′-Tetrahydroxybenzophenone. Needles + $2H_2O$ from H_2O. M.p. anhyd. 202°. Sol. EtOH, Et_2O, Me_2CO, AcOH. Less sol. H_2O. Spar. sol. C_6H_6. Insol. ligroin. Aq. sol. shows green fluor.

3′-Me ether: $C_{14}H_{12}O_5$. MW, 260. Yellowish needles from H_2O. M.p. 210°. Sol. EtOH, Et_2O. Spar. sol. H_2O. $FeCl_3$ ⟶ intense reddish-brown col.

Tetra-Me ether: prisms from EtOH. M.p. 126° (107°). Conc. H_2SO_4 ⟶ yellow sol.

M.L.B., D.R.P., 72,446.
Hoesch, v. Zarzecki, *Ber.*, 1917, **50**, 465.
Komarowski, Kostanecki, *Ber.*, 1894, **27**, 2000.
Mitter, Paul, *J. Indian Chem. Soc.*, 1931, **8**, 274.

2 : 4 : 3′ : 5′-Tetrahydroxybenzophenone. *Tetra-Me ether*: needles from C_6H_6–pet. ether. M.p. 73–4°. Sol. EtOH, Et_2O, $CHCl_3$, hot C_6H_6. Spar. sol. AcOH. Insol. pet. ether. Conc. H_2SO_4 ⟶ yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

2 : 5 : 2′ : 5′-Tetrahydroxybenzophenone. *Tetra-Me ether*: yellowish cryst. from EtOH. M.p. 109°. Sol. $CHCl_3$, C_6H_6. Less sol. EtOH, Et_2O, AcOH. *Oxime*: cryst. M.p. 134·5°. *Phenylhydrazone*: m.p. 170°.

Kauffmann, Grombach, *Ann.*, 1906, **344**, 74.
Kauffmann, Fritz, *Ber.*, 1908, **41**, 4425.
Staudinger, Schlenker, Goldstein, *Helv. Chim. Acta*, 1921, **4**, 341.

2 : 5 : 2′ : 6′-Tetrahydroxybenzophenone. Yellow needles from H_2O. M.p. 200–2° decomp.

Tetra-Et ether: $C_{21}H_{26}O_5$. MW, 358. Leaflets or needles from EtOH. M.p. 93–5°. Spar. sol. EtOH.

Tetra-acetyl: leaflets from EtOH. M.p. 118–19°.

Baeyer, *Ann.*, 1870, **155**, 259.
Herzig, *Monatsh.*, 1892, **13**, 412.

2 : 5 : 3′ : 4′-Tetrahydroxybenzophenone. *Tetra-Me ether*: prisms from EtOH.Aq. M.p. 101–2°. Conc. H_2SO_4 ⟶ orange sol.

König, Kostanecki, *Ber.*, 1906, **39**, 4030.

2 : 6 : 2′ : 6′-Tetrahydroxybenzophenone. *Tetra-Me ether*: plates from C_6H_6. M.p. 204°. Very sol. $CHCl_3$. Mod. sol. EtOH, AcOH. Spar. sol. Et_2O, C_6H_6.

Baeyer, *Ann.*, 1910, **372**, 130.

3 : 4 : 3′ : 4′-Tetrahydroxybenzophenone. Cryst. from H_2O. M.p. 227–8°.

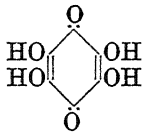
Tetra-Me ether: prisms from EtOH. M.p. 145°. Sol. EtOH, C_6H_6. Spar. sol. pet. ether. *Oxime*: cryst. from EtOH. M.p. 145°.

M.L.B., D.R.P., 72,446.
Kostanecki, Tambor, *Ber.*, 1906, **39**, 4027.
Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1661.

3 : 4 : 3′ : 5′-Tetrahydroxybenzophenone. *Tetra-Me ether*: needles from C_6H_6. M.p. 114–15°. Sol. EtOH, Et_2O, $CHCl_3$, hot C_6H_6. Spar. sol. AcOH. Insol. ligroin. Conc. H_2SO_4 ⟶ yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

Tetrahydroxy-*p*-benzoquinone



$C_6H_4O_6$ MW, 172

Bluish-black cryst. Very sol. EtOH, hot H_2O. Spar. sol. Et_2O, cold H_2O. Strong dibasic acid.

Di-Na salt: bluish-black cryst. with metallic green lustre.

Di-K salt: dark purple cryst. Very spar. sol. H_2O.

Diphenyl ether: $C_{18}H_{12}O_6$. MW, 324. Reddish-brown plates from EtOH. M.p. 276°. Sol. AcOH. Spar. sol. EtOH, hot $CHCl_3$. Insol. Et_2O, C_6H_6, CS_2, ligroin. *Di-Me ether*: $C_{20}H_{16}O_6$. MW, 352. Golden-yellow needles from C_6H_6–EtOH. M.p. 171°. Sol. $CHCl_3$. Mod. sol. EtOH, C_6H_6, AcOH. Spar. sol. Et_2O, CS_2. Insol. ligroin. *Di-Et ether*: $C_{22}H_{20}O_6$. MW, 380. Orange-yellow needles from EtOH. M.p. 128°. Sol. EtOH, $CHCl_3$. Spar. sol. Et_2O, AcOH.

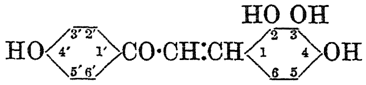
Tetraphenyl ether: $C_{30}H_{20}O_6$. MW, 476. Red prisms from C_6H_6. M.p. 229–30°. Spar. sol. CS_2, boiling AcOH. Very spar. sol. warm Me_2CO. Insol. EtOH, Et_2O, ligroin.

Diacetyl: yellow leaflets. M.p. 205°. Mod. sol. EtOH, Et_2O. Spar. sol. H_2O.

Jackson, Grindley, *Am. Chem. J.*, 1895, **17**, 647.
M.L.B., D.R.P., 368,741, (*Chem. Abstracts*, 1924, **18**, 991).
Maquenne, *Ann. chim. phys.*, 1887, **12**, 112.
Hoglan, Bartow, *J. Am. Chem. Soc.*, 1940, **62**, 2397.

1 : 2 : 3 : 4-Tetrahydroxybutane. *See* Erythritol *and* Threitol.

2 : 3 : 4 : 4′-Tetrahydroxychalkone (4-*Hydroxyphenyl* 2 : 3 : 4-*trihydroxystyryl ketone*)



$C_{15}H_{12}O_5$ MW, 272

Reddish-brown needles from EtOH.Aq. M.p. 117°.

Tetrabenzoyl: yellow cryst. from EtOH. M.p. 105°.

Russell, Todd, *J. Chem. Soc.*, 1937, 421.

2 : 4 : 6 : 4′-Tetrahydroxychalkone (4-*Hydroxyphenyl* 2 : 4 : 6-*trihydroxystyryl ketone*).

Orange prisms from EtOH.Aq. M.p. 205°.

Tetrabenzoyl: pale yellow cryst. from EtOH.

Russell, Todd, *J. Chem. Soc.*, 1937, 421.

2 : 4 : 2′ : 4′-Tetrahydroxychalkone (2 : 4-*Dihydroxyphenyl* 2 : 4-*dihydroxystyryl ketone*).

2 : 4 : 4′-*Tri-Me ether*: $C_{18}H_{18}O_5$. MW, 314. Yellow needles from EtOH. M.p. 157°. Conc. $H_2SO_4 \longrightarrow$ red sol. *Acetyl*: Pale yellow cryst. from EtOH. M.p. 110–12°.

4 : 2′ : 4′-*Tri-Me ether*: greenish-yellow needles from Me_2CO. M.p. 156°.

Tetra-Me ether: $C_{19}H_{20}O_5$. MW, 328. Yellowish needles from EtOH. M.p. 128°. Sol. Me_2CO, $CHCl_3$, CS_2, C_6H_6. Spar. sol. Et_2O, ligroin.

Bhalla, Rây, *J. Chem. Soc.*, 1933, 290.
Kostanecki, Lampe, Triulzi, *Ber.*, 1906, **39**, 92.
Kauffmann, Kieser, *Ber.*, 1913, **46**, 3797.

2 : 4 : 2′ : 5′-Tetrahydroxychalkone (2 : 5-*Dihydroxyphenyl* 2 : 4-*dihydroxystyryl ketone*).

2 : 4 : 5′-*Tri-Me ether*: orange-yellow needles from EtOH. M.p. 118°. Conc. $H_2SO_4 \longrightarrow$ red col. *Acetyl*: yellowish needles from EtOH. M.p. 87°.

Tetra-Me ether: cryst. from EtOH.Aq. M.p. 112°.

Tetrabenzoyl: yellow cryst. from EtOH. M.p. 137–9°.

Kauffmann, Kieser, *Ber.*, 1913, **46**, 3798.
Bonifazi, Kostanecki, Tambor, *Ber.*, 1906, **39**, 88.
Russell, Clark, *J. Am. Chem. Soc.*, 1939, **61**, 2651.

3 : 4 : 2′ : 4′-Tetrahydroxychalkone. *See* Butein.

3 : 4 : 2′ : 5′-Tetrahydroxychalkone (2 : 5-*Dihydroxyphenyl* 3 : 4-*dihydroxystyryl ketone*).

Dark orange micro-cryst. M.p. 225–7°.

3 : 5′-*Di-Me ether*: $C_{17}H_{16}O_5$. MW, 300. Orange-red leaflets from EtOH. M.p. 122–3°. Conc. $H_2SO_4 \longrightarrow$ yellowish-red sol.

Tetrabenzoyl: pale yellow cryst. from EtOH. M.p. 182–4°.

Milobedzka, Kostanecki, Lampe, *Ber.*, 1910, **43**, 2164.
Russell, Clark, *J. Am. Chem. Soc.*, 1939, **61**, 2651.

3 : 4 : 3′ : 4′-Tetrahydroxychalkone (3 : 4-*Dihydroxyphenyl* 3 : 4-*dihydroxystyryl ketone*).

Tetra-Me ether: yellow prisms from EtOH. M.p. 116° (110°). Conc. H_2SO_4 + AcOH $\longrightarrow$ dark red sol.

Kauffmann, Kieser, *Ber.*, 1913, **46**, 3798.
Perkin, Rây, Robinson, *J. Chem. Soc.*, 1926, 951.

3 : 5 : 3′ : 4′-Tetrahydroxychalkone (3 : 4-*Dihydroxyphenyl* 3 : 5-*dihydroxystyryl ketone*).

Tetra-Me ether: yellow needles from EtOH.Aq. M.p. 103–4°. Sol. EtOH, C_6H_6, warm ligroin. Insol. pet. ether. Conc. $H_2SO_4 \longrightarrow$ blood-red sol.

Mauthner, *J. prakt. Chem.*, 1920, **100**, 182.

2 : 2′ : 3′ : 4′-Tetrahydroxychalkone (2 : 3 : 4-*Trihydroxyphenyl* 2-*hydroxystyryl ketone*).

Yellow needles from 75% MeOH. M.p. 224–5° decomp. Very sol. EtOH, Me_2CO. Spar. sol. boiling xylene. Insol. H_2O, $CHCl_3$, C_6H_6. Conc. $H_2SO_4 \longrightarrow$ deep brown sol Alkalis $\longrightarrow$ deep brown sols.

2 : 3′ : 4′-*Tri-Me ether*: pale yellow needles from EtOH. M.p. 105°. Conc. $H_2SO_4 \longrightarrow$ orange-red sol. *Acetyl*: needles from EtOH. M.p. 88°.

Ellison, *J. Chem. Soc.*, 1927, 1723.
Cohen, Kostanecki, *Ber.*, 1904, **37**, 2628.

2 : 2′ : 4′ : 5′-Tetrahydroxychalkone (2 : 4 : 5-*Trihydroxyphenyl* 2-*hydroxystyryl ketone*).

2′ : 4′ : 5′-*Tri-Me ether*: green needles from EtOH. M.p. 159–60° decomp. Conc. $H_2SO_4 \longrightarrow$ orange sol. *Acetyl*: golden needles from EtOH.Aq. M.p. 121°.

Reigrodski, Tambor, *Ber.*, 1910, **43**, 1967.

2 : 2′ : 4′ : 6′-Tetrahydroxychalkone (2 : 4 : 6-*Trihydroxyphenyl* 2-*hydroxystyryl ketone*).

2 : 4′ : 6′-*Tri-Me ether*: yellow cryst. from boiling MeOH. M.p. 106–8°. Conc. $H_2SO_4 \longrightarrow$ orange-red col.

Bargellini, Peratoner, *Gazz. chim. ital.*, 1919, **49**, ii, 67.

3 : 2′ : 3′ : 4′-Tetrahydroxychalkone (2 : 3 : 4-*Trihydroxyphenyl* 3-*hydroxystyryl ketone*).

Yellow needles from 80% MeOH. M.p. 219–20°. Conc. $H_2SO_4 \longrightarrow$ deep orange sol. Alkalis $\longrightarrow$ deep brown sols.

3 : 3′ : 4′-*Tri-Me ether*: yellow needles from EtOH. M.p. 127–8°. Conc. $H_2SO_4 \longrightarrow$

orange-yellow sol. *Acetyl*: yellowish needles from EtOH. M.p. 80–1°.

Ellison, *J. Chem. Soc.*, 1927, 1723.
Kostanecki, Schleifenbaum, *Ber.*, 1904, **37**, 2631.

4 : 2′ : 3′ : 4′ - Tetrahydroxychalkone (2:3:4-*Trihydroxyphenyl 4-hydroxystyryl ketone*).

3′ : 4′-*Di-Me ether*: yellow needles from EtOH.Aq. M.p. 106°.

4 : 3′ : 4′-*Tri-Me ether*: yellow plates. M.p. 131–2°. Conc. H_2SO_4 ⟶ orange-yellow sol. *Acetyl*: yellowish prisms from EtOH.Aq. M.p. 89–90°.

Tetra-Me ether: pale yellow needles from EtOH. M.p. 94°.

Russell, *J. Chem. Soc.*, 1934, 220.
Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.
Kostanecki, Schreiber, *Ber.*, 1905, **38**, 2749.

4 : 2′ : 4′ : 5′ - Tetrahydroxychalkone (2:4:5-*Trihydroxyphenyl 4-hydroxystyryl ketone*).

4 : 4′ : 5′-*Tri-Me ether*: red needles from EtOH.Aq. M.p. 130°. Sol. EtOH, C_6H_6. Conc. H_2SO_4 ⟶ orange-yellow sol.

Tetra-Me ether: yellow needles from EtOH. M.p. 123–4°. Conc. H_2SO_4 ⟶ intense red sol.

Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.
Bargellini, Aureli, *Atti accad. Lincei*, 1911, **20**, II, 123.
Bargellini, Aurutin, *Gazz. chim. ital.*, 1910, **40**, ii, 346.

4 : 2′ : 4′ : 6′ - Tetrahydroxychalkone (2:4:6-*Trihydroxyphenyl 4-hydroxystyryl ketone*).

4′ : 6′-*Di-Me ether*: orange-red needles from C_6H_6. M.p. 188°. Conc. H_2SO_4 ⟶ dark yellow sol. *Diacetyl*: pale yellow needles from MeOH. M.p. 147°.

4 : 4′ : 6′-*Tri-Me ether*: yellow needles from EtOH. M.p. 113°. Conc. H_2SO_4 ⟶ orange sol. *Acetyl*: yellowish leaflets from EtOH. M.p. 120°.

2′ : 4′ : 6′-*Tri-Me ether*: golden-yellow cryst. from MeOH. M.p. 195–6°. Conc. H_2SO_4 ⟶ orange-red sol. *Acetyl*: yellow cryst. from MeOH. M.p. 108°.

Tetra-Me ether: pale yellow cryst. from EtOH.Aq. M.p. 119–21°. Conc. H_2SO_4 ⟶ orange-red sol.

4-*Carbomethoxyl*: yellow cryst. from MeOH.Aq. M.p. 166°.

Mosimann, Tambor, *Ber.*, 1916, **49**, 1701.
Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.
Bargellini, *Gazz. chim. ital.*, 1914, **44**, ii, 424.
Kostanecki, Tambor, *Ber.*, 1904, **37**, 792.

2′ : 3′ : 4′ : 6′ - Tetrahydroxychalkone (2 : 3 : 4 : 6-*Tetrahydroxyphenyl styryl ketone*).

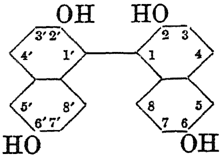
Tetra-Me ether: yellowish needles from EtOH.Aq. M.p. 74–5°. Sol. C_6H_6. Conc. H_2SO_4 ⟶ orange-red sol.

Bargellini, Bini, *Gazz. chim. ital.*, 1911, **41**, ii, 18.

1 : 3 : 4 : 5 - Tetrahydroxycyclohexane-1-carboxylic Acid.

See Quinic Acid.

2 : 6 : 2′ : 6′ - Tetrahydroxy - 1 : 1′ - dinaphthyl



$C_{20}H_{14}O_4$ MW, 318

M.p. 318–20°. Autoxidises readily, especially in alk. sol.

Ioffe, Kusnetzow, *Chem. Zentr.*, 1936, I, 2935.

3 : 4 : 3′ : 4′ - Tetrahydroxy - 1 : 1′ - dinaphthyl.

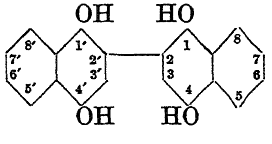
Needles from 30% AcOH. M.p. 205–10° decomp. (176–8°). Sol. AcOH. Spar. sol. Et_2O, C_6H_6, CS_2. Insol. H_2O.

Tetra-Me ether: $C_{24}H_{22}O_4$. MW, 374. Leaflets from EtOH. M.p. 145–7°. B.p. 200–20°/0·67 mm. Sol. most org. solvents. Spar. sol. EtOH, pet. ether. Conc. H_2SO_4 ⟶ bluish-violet col.

Tetra-acetyl: needles from AcOH. M.p. 165–6° decomp. Sol. usual solvents.

Korn, *Ber.*, 1884, **17**, 3025.
Strauss, Bernoully, Mautner, *Ann.*, 1925, **444**, 190.

1 : 4 : 1′ : 4′ - Tetrahydroxy - 2 : 2′ - dinaphthyl



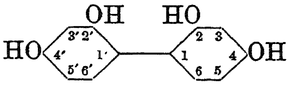
$C_{20}H_{14}O_4$ MW, 318

Needles from AcOH. Blackens at 205°. Spar. sol. cold EtOH. Insol. H_2O. Alc. sol. turns violet in air.

Tetra-acetyl: needles from AcOH. M.p. 226–7°.

Meldola, Hughes, *J. Chem. Soc.*, 1890, **57**, 632.
See also Liebermann, Schlossberg, *Ber.* 1899, **32**, 546.

2 : 4 : 2′ : 4′-Tetrahydroxydiphenyl



$C_{12}H_{10}O_4$ MW, 218

Needles from boiling H_2O. M.p. 226–7°. Sol. EtOH, Et_2O, Me_2CO, AcOEt.

Tetra-Me ether: $C_{16}H_{18}O_4$. MW, 274. Cryst. from MeOH. M.p. 93°.

Tetra-acetyl: needles from Et_2O. M.p. 120°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, AcOH, C_6H_6.

Meyer, Desamari, *Ber.*, 1909, **42**, 2823.
Bayer, D.R.P., 90,341.

2 : 4 : 2′ : 5′-Tetrahydroxydiphenyl (*Resorcylhydroquinone*).

Needles from C_6H_6. M.p. 131° under CO_2. Very sol. H_2O, EtOH, Et_2O. Mod. sol. C_6H_6, ligroin. Spar. sol. CS_2. Alkalis ⟶ olive-green sols.

Tetra-acetyl: needles from ligroin. M.p. 120° uuder CO_2. Sol. $CHCl_3$, AcOEt, C_6H_6. Mod. sol. EtOH, Et_2O, ligroin, hot H_2O. Insol. cold NaOH.

Pummerer, Huppmann, *Ber.*, 1927, **60**, 1446.

2 : 4 : 3′ : 4′-Tetrahydroxydiphenyl.
See Sappanin.

2 : 5 : 2′ : 5′-Tetrahydroxydiphenyl (*Dihydroquinone*).

Colourless prisms. M.p. 237° decomp. Sol. EtOH, Et_2O, Me_2CO, hot H_2O. Spar. sol. $CHCl_3$, C_6H_6. $FeCl_3$ ⟶ red col.

Tetra-Me ether: $C_{16}H_{18}O_4$. MW, 274. M.p. 104°. Sol. AcOH, C_6H_6, hot EtOH. Spar. sol. Et_2O.

Barth, Schreider, *Monatsh.*, 1884, **5**, 590.
Ullmann, Loewenthal, *Ann.*, 1904, **332**, 69.

3 : 4 : 3′ : 4′-Tetrahydroxydiphenyl (*Dicatechol*).

Cryst. from H_2O. M.p. 229–30°.

Tetra-Me ether: diveratrol. Needles from MeOH.Aq. M.p. 133°. Conc. H_2SO_4 ⟶ golden-yellow sol. ⟶ olive-green col. on warming.

Seer, Karl, *Monatsh.*, 1913, **34**, 647.
Späth, Gibian, *Monatsh.*, 1930, **55**, 347.

3 : 5 : 3′ : 5′-Tetrahydroxydiphenyl (*Diresorcinol*).

Needles + $2H_2O$ from hot H_2O. M.p. anhyd. 310°. Mod. sol. hot H_2O. Prac. insol. AcOH. Zn dust dist. ⟶ diphenyl. $FeCl_3$ ⟶ light blue col.

Tetra-Me ether: white cryst. M.p. 108°.

Tetra-Et ether: $C_{20}H_{26}O_4$. MW, 330. Leaflets. M.p. 110°. Sol. Et_2O.

Tetra-acetyl: m.p. 157–9°.

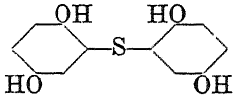
Benedikt, Julius, *Monatsh.*, 1884, **5**, 177.
v. Friedrichs, *Chem. Zentr.*, 1916, I, 975.

2 : 6 : 2′ : 6′-Tetrahydroxydiphenyl.

Tetra-Me ether: cryst. from Me_2CO.Aq. M.p. 175–6°.

v. Arendonk, Cupery, Adams, *J. Am. Chem. Soc.*, 1933, **55**, 4227.

2 : 5 : 2′ : 5′-Tetrahydroxydiphenyl sulphide



$C_{12}H_{10}O_4S$ MW, 250

Cryst. M.p. 227–9°.

Tetra-Me ether: $C_{16}H_{18}O_4S$. MW, 306. Amorph. M.p. 97–100°. Sol. Me_2CO, hot EtOH.

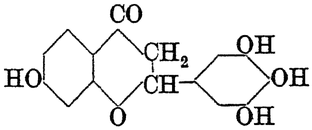
Badische, D.R.P., 175,070, (*Chem. Zentr.*, 1906, I, 1466).
Smiles, Le Rossignol, *J. Chem. Soc.*, 1908, **93**, 760.

2 : 3 : 2′ : 3′-Tetrahydroxydipropyl Ether.
See Diglycerol.

5 : 6 : 7 : 4′-Tetrahydroxyflavanone.
See Isocarthamidin.

5 : 7 : 8 : 4′-Tetrahydroxyflavanone.
See Carthamidin.

7 : 3′ : 4′ : 5′-Tetrahydroxyflavanone

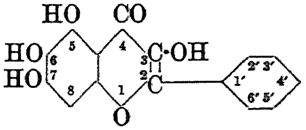


$C_{15}H_{12}O_6$ MW, 288

Tetra-Me ether: $C_{19}H_{20}O_6$. MW, 344. Needles from EtOH. M.p. 148–9°. Conc. H_2SO_4 ⟶ yellow sol.

Dean, Nierenstein, *J. Am. Chem. Soc.*, 1925, **47**, 1681.

3 : 5 : 6 : 7-Tetrahydroxyflavone (5 : 6 : 7-*Trihydroxyflavonol*)



$C_{15}H_{10}O_6$ MW, 286

Cryst. from EtOH. M.p. 250–1°.

Tetra-acetyl: m.p. 186–8°.

Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23**, 23, (*Chem. Abstracts*, 1946, **40**, 5050).

3 : 5 : 7 : 8-Tetrahydroxyflavone (5 : 7 : 8-*Trihydroxyflavonol*).

M.p. 231–3°. Deep red ppt. with $Pb(OAc)_2$ in EtOH. $FeCl_3$ ⟶ red col.

Tetra-Me ether: m.p. 156–8°.

Tetra-acetyl: m.p. 169–70°.

Rao, Seshadri, *Proc. Indian Acad. Sci.*, 1945, **22**, 157, (*Chem. Abstracts*, 1946, **40**, 1832).

3 : 6 : 7 : 8-Tetrahydroxyflavone (6 : 7 : 8-*Trihydroxyflavonol*).

Cryst. from EtOH. M.p. 280°. Conc. H_2SO_4 ⟶ yellow col.

Tetra-Me ether : yellow cryst. from MeOH. M.p. 116–17°.

Tetra-acetyl : cryst. from EtOH or AcOH. M.p. 220–21°.

Bargellini, Oliverio, *Ber.*, 1942, **75**, 2083.

5 : 6 : 7 : 8-Tetrahydroxyflavone.

M.p. 275–7°.

Tetra-Me ether : m.p. 117–18°.

Tetra-acetyl : m.p. 215–17°.

Murti, Rao, Seshadri, *Proc. Indian Acad. Sci.*, 1947, **26**, 183, (*Chem. Abstracts*, 1948, **42**, 5908).

3 : 5 : 6 : 4′-Tetrahydroxyflavone (5 : 6 : 4′-*Trihydroxyflavonol*).

Sinters at 294°. M.p. 305°. Alkalis ⟶ reddish-brown col.

Tetra-Me ether : m.p. 147–8°.

Rajagopalan, Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23**, 97, (*Chem. Abstracts*, 1947, **41**, 120).

3 : 5 : 7 : 2′-Tetrahydroxyflavone.

See Datiscetin.

3 : 5 : 7 : 4′-Tetrahydroxyflavone.

See Kæmpferol.

3 : 6 : 7 : 4′-Tetrahydroxyflavone (6 : 7 : 4′-*Trihydroxyflavonol*).

Yellow plates. Does not melt below 320°.

Tetra-Me ether : colourless rectangular plates. M.p. 164–5°. Insol. alkalis.

Tetra-acetyl : m.p. 193–5°.

Rao, Row, Seshadri, *Proc. Indian Acad. Sci.*, 1945, **22**, 297, (*Chem. Abstracts*, 1946, **40**, 3756).

3 : 7 : 8 : 2′-Tetrahydroxyflavone (7 : 8 : 2′-*Trihydroxyflavonol*).

Pale yellow needles. M.p. 298° decomp. NaOH ⟶ reddish-yellow sol.

7 : 8 : 2′-*Tri-Me ether* : $C_{18}H_{16}O_6$. MW, 328. Cryst. from EtOH. M.p. 212–14°. NaOH ⟶ yellow sol. Conc. H_2SO_4 ⟶ greenish-yellow sol. *Acetyl* : needles from EtOH.Aq. M.p. 138–9°.

Cohen, Kostanecki, *Ber.*, 1904, **37**, 2630.

3 : 7 : 8 : 3′-Tetrahydroxyflavone (7 : 8 : 3′-*Trihydroxyflavonol*).

Pale yellow needles from EtOH.Aq. M.p. 260°. Conc. H_2SO_4 ⟶ greenish-yellow sol. Alkalis ⟶ brownish-yellow sols.

7 : 8 : 3′-*Tri-Me ether* : pale yellow needles. M.p. 188–9°. Spar. sol. EtOH. Conc. H_2SO_4 ⟶ pale yellow sol. *Acetyl* : needles from EtOH.Aq. M.p. 165°.

Tetra-acetyl : needles from EtOH.Aq. M.p. 166–7°.

Kostanecki, Schleifenbaum, *Ber.*, 1904, **37**, 2633.

3 : 7 : 8 : 4′-Tetrahydroxyflavone (7 : 8 : 4′-*Trihydroxyflavonol*).

Pale yellow needles + H_2O from EtOH. M.p. anhyd. 319° decomp. Dil. NaOH ⟶ orange-red sol. Conc. H_2SO_4 ⟶ yellow sol. with weak green fluor.

7 : 8 : 4′-*Tri-Me ether* : pale yellow needles from EtOH. M.p. 198°. Conc. H_2SO_4 ⟶ pale yellow sol. *Acetyl* : needles from EtOH.Aq. M.p. 157°.

Tetra-acetyl : needles from EtOH.Aq. M.p. 175°.

Kostanecki, Schreiber, *Ber.*, 1905, **38**, 2751.

5 : 6 : 7 : 4′-Tetrahydroxyflavone.

See Scutellarein.

5 : 7 : 8 : 4′-Tetrahydroxyflavone.

Yellow needles from AcOH.Aq. M.p. 247–8°. Alkalis ⟶ reddish-brown sols. $FeCl_3$ ⟶ greenish-brown col.

5 : 8 : 4′-*Tri-Me ether* : yellow cryst. from EtOH. M.p. 279–80° (258°). *Acetyl* : m.p. 194·5°.

Tetra-Me ether : needles. M.p. 207–8°.

Tetra-acetyl : needles from EtOH. M.p. 252–3°.

Furukawa, Tamaki, *Chem. Abstracts*, 1932, **26**, 142.

Hattori, *Acta Phytochim.*, 1931, **5**, 219.

Weselý, *Monatsh.*, 1930, **56**, 97.

6 : 7 : 8 : 4′-Tetrahydroxyflavone.

Yellow cryst. from EtOH. M.p. 250° decomp. Conc. H_2SO_4 ⟶ yellow col.

Tetra-Me ether : yellow cryst. from EtOH. M.p. 175–6°.

Tetra-acetyl : cryst. from AcOH. M.p. 249°. Very sol. EtOH.

Bargellini, Oliverio, *Ber.*, 1942, **75**, 2083.

Sastri, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23**, 134, (*Chem. Abstracts*, 1947, **41**, 120).

3 : 6 : 2′ : 4′-Tetrahydroxyflavone (6:2′:4′-*Trihydroxyflavonol*).

Pale yellow needles + H_2O from EtOH.Aq. M.p. anhyd. 285°. Dil. NaOH ⟶ greenish-yellow sol. with greenish fluor. Conc. H_2SO_4 ⟶ pale yellow sol. with weak green fluor.

6 : 2′ : 4′-*Tri-Me ether* : pale yellow cryst. from EtOH or C_6H_6. M.p. 193°. Conc. H_2SO_4 ⟶ greenish-yellow sol. with light green fluor. *Acetyl* : prismatic needles from EtOH.Aq. M.p. 162°.

Tetra-acetyl : needles from EtOH.Aq. M.p. 163°.

Bonifazi, Kostanecki, Tambor, *Ber.*, 1906, **39**, 90.

3 : 6 : 3′ : 4′-Tetrahydroxyflavone (*6:3′:4′-Trihydroxyflavonol*).

Yellow needles from EtOH. M.p. 335° decomp. Alc. sol. shows greenish fluor. NaOH ⟶ reddish-yellow sol. Conc. H_2SO_4 ⟶ orange sol. with greenish fluor.

6 : 3′ : 4′-*Tri-Me ether*: yellowish needles from EtOH. M.p. 189–90°. Conc. H_2SO_4 ⟶ yellow sol. with greenish fluor. *Acetyl*: needles from EtOH.Aq. M.p. 140–1°.

Tetra-acetyl: needles from EtOH or AcOH–EtOH. M.p. 197–8°. Spar. sol. EtOH.

Kostanecki, Kugler, *Ber.*, 1904, **37**, 781.
Auwers, Pohl, *Ann.*, 1914, **405**, 287.

3 : 7 : 2′ : 4′-Tetrahydroxyflavone.

See Resomorin.

3 : 7 : 3′ : 4′-Tetrahydroxyflavone.

See Fisetin.

5 : 7 : 2′ : 4′-Tetrahydroxyflavone.

Needles from EtOH or AcOH. M.p. 332–5° (277°) decomp. Conc. H_2SO_4 ⟶ pale yellow sol. with violet-blue fluor.

2′ : 4′-*Di-Me ether*: pale yellow needles from AcOH.Aq. + HCl. M.p. 258–9°. Mod. sol. boiling EtOH. Spar. sol. Et_2O. Insol. H_2O. Alc. $FeCl_3$ ⟶ weak greenish-brown col. Alkalis ⟶ bright yellow sols. Conc. H_2SO_4 ⟶ pale yellow sol. with blue fluor.

Robinson, Venkataraman, *J. Chem. Soc.*, 1929, 65.

5 : 7 : 3′ : 4′-Tetrahydroxyflavone.

See Luteolin.

7 : 8 : 3′ : 4′-Tetrahydroxyflavone.

Yellow needles from EtOH.Aq. M.p. 309–10° decomp. Conc. H_2SO_4 ⟶ pale yellow col. with no fluor. NaOH ⟶ orange-red sol. Alc. $FeCl_3$ ⟶ dark green col.

Tetra-Me ether: yellow needles from EtOH. M.p. 198–9°.

Tetra-acetyl: needles from EtOH. M.p. 218°.

Badhwar, Kang, Venkataraman, *J. Chem. Soc.*, 1932, 1109.

6 : 3′ : 4′ : 5′-Tetrahydroxyflavone.

Yellow needles from EtOH.Aq. M.p. 347°. Conc. H_2SO_4 ⟶ bright yellow sol. with no fluor. NaOH ⟶ bright red sol. Alc. $FeCl_3$ ⟶ intense green col.

3′ : 4′ : 5′-*Tri-Me ether*: pale orange plates from EtOH.Aq. M.p. 232–3°. No col. with $FeCl_3$. Conc. H_2SO_4 ⟶ bright yellow sol. with weak green fluor. *Acetyl*: needles from EtOH. M.p. 185°.

Tetra-acetyl: needles from EtOH–AcOH. M.p. 258–9°.

Chadha, Venkataraman, *J. Chem. Soc.*, 1933, 1075.

7 : 2′ : 4′ : 6′-Tetrahydroxyflavone.

Yellow plates from EtOH. M.p. 240°. Spar. sol. $CHCl_3$. Conc. H_2SO_4 ⟶ yellow sol. with greenish fluor.

Cullinane, Algar, Ryan, *Chem. Abstracts*, 1929, **23**, 4472.

7 : 3′ : 4′ : 5′-Tetrahydroxyflavone.

Needles + H_2O from EtOH.Aq. M.p. 340° decomp. Mod. sol. hot EtOH. NaOH ⟶ orange-red sol. Conc. H_2SO_4 ⟶ yellow sol. with weak greenish-yellow fluor.

Tetra-Me ether: needles. M.p. 191–2°. Mod. sol. boiling EtOH with violet fluor. Conc. H_2SO_4 ⟶ yellowish sol. with weak green fluor.

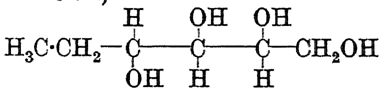
Tetra-acetyl: leaflets from EtOH. M.p. 215°.

Kostanecki, Plattner, *Ber.*, 1902, **35**, 2546.

1 : 3 : 4 : 5 - Tetrahydroxyhexahydrobenzoic Acid.

See Quinic Acid.

1 : 2 : 3 : 4-Tetrahydroxyhexane (*Hexantetrol*-1 : 2 : 3 : 4)



$C_6H_{14}O_4$ MW, 150

D-*Arabo*-.

Cryst. from EtOH–Me_2CO. M.p. 122°. $[\alpha]_D^{18}$ + 4·2°.

Karrer, Davis, *Helv. Chim. Acta*, 1948, **31**, 1611.

1 : 2 : 5 : 6-Tetrahydroxyhexane (*Hexantetrol*-1 : 2 : 5 : 6)

$HO{\cdot}CH_2{\cdot}CH(OH){\cdot}[CH_2]_2{\cdot}CH(OH){\cdot}CH_2OH$

$C_6H_{14}O_4$ MW, 150

Cryst. from EtOH–Et_2O or EtOH–AcOEt. M.p. 95° (98°).

Tetra-acetyl: cryst. from pet. ether–Et_2O. M.p. 78°.

Wagner, *Ber.*, 1888, **21**, 3344.
Milas, *J. Am. Chem. Soc.*, 1937, **59**, 2345.
Haworth, Jones, Wiggins, *J. Chem. Soc.*, 1945, 1.

1 : 3 : 4 : 6-Tetrahydroxyhexane (*Hexantetrol*-1 : 3 : 4 : 6)

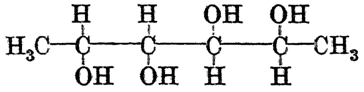
$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2OH$

$C_6H_{14}O_4$ MW, 150

Elongated prisms from EtOH–pet. ether. M.p. 122°. Very sol. H_2O, EtOH.

Wolfrom, Lew, Goepp, *J. Am. Chem. Soc.*, 1946, **68**, 1443.

2 : 3 : 4 : 5-Tetrahydroxyhexane (*Hexantetrol*-2 : 3 : 4 : 5)



$C_6H_{14}O_4$ MW, 150

D-*Mannito-*.
Cryst. from EtOH–Et_2O. M.p. 148° (150°). $[\alpha]_D^{15}$ − 17·6° in $CHCl_3$ ($[\alpha]_D^{25}$ + 28·6°).
Tetra-acetyl: cryst. from ligroin. M.p. 79°.

Micheel, *Ann.*, 1932, **496**, 77.
Hamamura, *Chem. Abstracts*, 1935, **29**, 1070.

5 : 7 : 3′ : 4′-Tetrahydroxyisoflavone.
See Orobol.

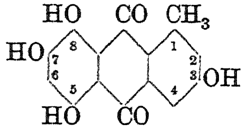
Tetrahydroxyisovaleraldehyde.
See Apiose.

Tetrahydroxyisovaleric Acid.
See Apionic Acid.

Tetrahydro-xylene.
See Dimethylcyclohexene.

Tetrahydroxymethane.
See Orthocarbonic Acid.

3 : 5 : 7 : 8 - Tetrahydroxy - 1 - methyl - anthraquinone



$C_{15}H_{10}O_6$ MW, 286

Light red needles from EtOH or Py. Sol. EtOH, AcOH, Py. Spar. sol. C_6H_6.
Tetra-acetyl: m.p. 185–6°.

Dimroth, Fick, *Ann.*, 1916, **411**, 325.

1 : 3 : 5 : 8 - Tetrahydroxy - 2 - methyl - anthraquinone.
Yellowish-red plates from 50% AcOH. M.p. 276–7°.
Tetra-acetyl: yellow plates from AcOH. M.p. 223°.

Charlesworth, Robinson, *J. Chem. Soc.*, 1934, 1531.

1 : 4 : 5 : 7 - Tetrahydroxy - 2 - methyl - anthraquinone.
See Catenarin.

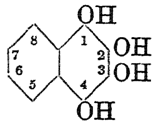
1 : 4 : 5 : 8 - Tetrahydroxy - 2 - methyl - anthraquinone.
See Cynodontin.

1 : 5 : 6 : 7 - Tetrahydroxy - 2- methyl - anthraquinone (*Areolatin, copareolatin*).
Found in *Coprosma areolata*. Cryst. from AcOH. Decomp. above 298°. Insol. H_2O. Very sol. Py, Me_2CO, dioxan. Conc. H_2SO_4 ⟶ deep reddish-purple col. NaOH ⟶ reddish-brown col. NH_4OH ⟶ greenish-blue col.
Tetra-acetyl: long yellow needles from AcOH. M.p. 238°.
Tetra-benzoyl: plates from AcOH. M.p. 281°.
Tetra-Me ether: needles from AcOH. M.p. 175°.

Briggs, Craw, Dacre, *J. Chem. Soc.*, 1948, 568.
Briggs, Nicholls, Paterson, *J. Chem. Soc.*, 1952, 1718.

Tetrahydroxymethyl-methane.
See Pentaerythritol.

1 : 2 : 3 : 4 - Tetrahydroxynaphthalene (*Leucoisonaphthazarin*)



$C_{10}H_8O_4$ MW, 192

Cryst. from C_6H_6, leaflets from Et_2O–pet. ether. M.p. 225°. Sol. H_2O, most org. solvents. Spar. sol. C_6H_6, pet. ether. Easily decomp.
Tetra-acetyl: needles from AcOH. M.p. 220°. Mod. sol. EtOH, C_6H_6.

Zincke, Ossenbeck, *Ann.*, 1899, **307**, 16.
Leeds, *J. Am. Chem. Soc.*, 1880, **2**, 285.

1 : 2 : 4 : 5(or 8)-Tetrahydroxynaphthalene.
Tetra-acetyl: obtained by action of Ac_2O–H_2SO_4 on juglone. Prisms from EtOH. M.p. 154°.

Mylius, *Ber.*, 1885, **18**, 463.
Fieser, Dunn, *J. Am. Chem. Soc.*, 1937, **59**, 1016.

1 : 2 : 4 : 6-Tetrahydroxynaphthalene.
Tetra-acetyl: leaflets from AcOH. M.p. 181–2°.

Dimroth, Kerkovius, *Ann.*, 1913, **399**, 39.

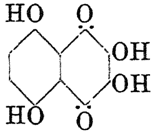
1 : 2 : 4 : 7-Tetrahydroxynaphthalene.
Tetra-acetyl: cryst. from ligroin. M.p. 140–1°.

Dimroth, Kerkovius, *Ann.*, 1913, **399**, 39.

1 : 4 : 5 : 8 - Tetrahydroxynaphthalene (*Leuconaphthazarin*).
Yellow plates or prisms from HCl. M.p. 153–4°. Mod. sol. EtOH, C_6H_6. Spar. sol. H_2O, pet. ether. Turns red in air.
Tetra-Me ether: $C_{14}H_{16}O_4$. MW, 248. Cryst. from AcOH. M.p. about 170°. Sol. AcOH, toluene, xylene.
1 : 4-*Diacetyl*: plates from AcOEt. M.p. 241–3°. Very sol. Me_2CO. Sol. EtOH. Insol. $CHCl_3$, xylene.
Tetra-acetyl: leaflets from AcOH. M.p. 277–9° decomp. Sol. hot AcOH. Spar. sol. other solvents.

Zincke, Schmidt, *Ann.*, 1895, **286**, 37.
Badische, D.R.P., 129,074, (*Chem. Zentr.*, 1902, I, 691).
Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1658.
Wheeler, Edwards, *J. Am. Chem. Soc.*, 1916, **38**, 387; 1917, **39**, 2465.

2 : 3 : 5 : 8-Tetrahydroxy-1 : 4-naphthoquinone



$C_{10}H_6O_6$ MW, 222

Cryst. from AcOH. M.p. 265°.

Tetra-acetyl: m.p. 207°.

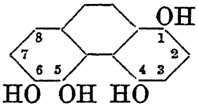
5 : 8-*Di-Me ether*: m.p. 129°.

Kuroda, *Chem. Abstracts*, 1940, **34**, 6215.
Wallenfels, Gauhe, *Ber.*, 1942, **75**, 413.

2 : 3 : 4 : 6-Tetrahydroxyphenacyl Alcohol.

See Gossypitol.

1 : 4 : 5 : 6-Tetrahydroxyphenanthrene



$C_{14}H_{10}O_4$ MW, 242

Tetra-Me ether: $C_{18}H_{18}O_4$. MW, 298. Light brown needles from MeOH. M.p. 118–20°. *Picrate*: dark chocolate needles from EtOH. M.p. 158°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 1486.

2 : 3 : 5 : 6-Tetrahydroxyphenanthrene.

2 : 6-*Di-Me ether*: sinomenol. $C_{16}H_{14}O_4$. MW, 270. M.p. 176°. *Diacetyl*: m.p. 206°.

Tetra-Me ether: cryst. from EtOH. M.p. 124–5°. *Picrate*: m.p. 123–5°.

Kondo, Ochiai, *Chem. Abstracts*, 1928, **22**, 4532.
Goto, *Chem. Abstracts*, 1927, **21**, 1654.

Tetrahydroxyphenyl styryl Ketone.

See 2′ : 3′ : 4′ : 6′-Tetrahydroxychalkone.

8 : 9 : 11 : 12-Tetrahydroxystearic Acid.

See Sativic Acid.

3 : 4 : 3′ : 5′ - Tetrahydroxystilbene 4 - methyl Ether.

See Rhapontigenin.

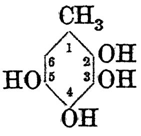
Tetrahydroxysuccinic Acid.

See Diketosuccinic Acid.

Tetrahydroxytetramethylmethane.

See Pentaerythritol.

2 : 3 : 4 : 5 - Tetrahydroxytoluene (5-*Methylapionol*)



$C_7H_8O_4$ MW, 156

Cryst. from toluene. M.p. 170–1°. Sol. H_2O, EtOH. Spar. sol. C_6H_6, toluene. NaOH ⟶ intense green col. ⟶ blue on shaking in air.

4-*Me ether*: $C_8H_{10}O_4$. MW, 170. *Triacetyl*: m.p. 91–2°.

Tetra-Me ether: $C_{11}H_{16}O_4$. MW, 212. M.p. 51–2°.

Tetra-acetyl: cryst. from MeOH. M.p. 132–3°. Sol. most org. solvents.

Thiele, Winter, *Ann.*, 1900, **311**, 352.
Erdtman, *Chem. Abstracts*, 1934, **28**, 1337.

2 : 3 : 4 : 6-Tetrahydroxytoluene.

4-*Me ether*: cryst. Oxidises at 134°. Sol. H_2O, AcOEt. Mod. sol. EtOH, Et_2O. Insol. C_6H_6, ligroin. *Triacetyl*: cryst. from AcOEt. M.p. 174°.

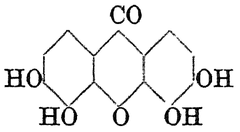
Konya, *Monatsh.*, 1900, **21**, 430.

2 : 3 : 5 : 6-Tetrahydroxytoluene.

Tetra-acetyl: needles from EtOH. M.p. 198°.

Fichter, *Ann.*, 1908, **361**, 401.

1 : 2 : 8 : 9-Tetrahydroxyxanthone



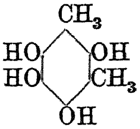
$C_{13}H_8O_6$ MW, 260

Light brown cryst. powder. Sol. EtOH, Me_2CO. Spar. sol. hot H_2O. Insol. $CHCl_3$, C_6H_6. NaOH ⟶ yellowish-brown sol.

Tetra-acetyl: cryst. from C_6H_6. M.p. 237°.

Buchka, *Ann.*, 1881, **209**, 270.

2 : 4 : 5 : 6-Tetrahydroxy-*m*-xylene



$C_8H_{10}O_4$ MW, 170

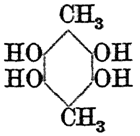
Needles from Et_2O-ligroin. M.p. 189° (rapid heat.). Sol. H_2O, EtOH, Et_2O. Insol. ligroin. Conc. H_2SO_4 ⟶ green col. ⟶ bluish-violet on dilution.

4-*Me ether*: prisms from C_6H_6. M.p. 125°. *Triacetyl*: cryst. from EtOH. M.p. 76°. Sol. EtOH, Et_2O, C_6H_6, ligroin.

Tetra-acetyl: needles from boiling EtOH. M.p. 154°. Sol. Et_2O. Insol. cold H_2O, EtOH.

Brunnmayr, *Monatsh.*, 1900, **21**, 10.
Bosse, *ibid.*, 1028.

2 : 3 : 5 : 6-Tetrahydroxy-*p*-xylene



$C_8H_{10}O_4$ MW, 170

2 : 5-*Diacetyl*: needles from AcOH. M.p. 223°.

Tetra-acetyl: needles from EtOH. M.p. 242°.

Fichter, *Ann.*, 1908, **361**, 378.

sym.-Tetraiodoacetone

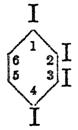
$I_2CH{\cdot}CO{\cdot}CHI_2$

$C_3H_2OI_4$ MW, 562

Yellow needles. M.p. 142°. Very sol. Me_2CO.

Angeli, Levi, *Gazz. chim. ital.*, 1893, **23**, ii, 97.
Lederer, D.R.P., 95,440.

1 : 2 : 3 : 4-Tetraiodobenzene



$C_6H_2I_4$ MW, 582

Prisms from CS_2 or Et_2O–AcOH. M.p. 136° (114°). Sublimes. Sol. EtOH, Et_2O, $CHCl_3$.

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3353.
Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 688.

1 : 2 : 3 : 5-Tetraiodobenzene.

Cryst. from AcOH or Et_2O. M.p. 148°. Sublimes. Sol. boiling AcOH. Spar. sol. EtOH, Et_2O, $CHCl_3$.

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3350.

1 : 2 : 4 : 5-Tetraiodobenzene.

Needles from Et_2O, prisms from C_6H_6. M.p. 254° (165°). Sol. AcOH, CS_2. Very spar. sol. EtOH, Et_2O. Sublimes in vacuo.

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3352.
Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 687.

Tetraiodobenzoquinone.
See Iodanil.

Tetraiodoethylene (*Periodoethylene*)

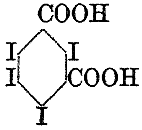
$I_2C{:}CI_2$

C_2I_4 MW, 532

Lemon-yellow leaflets from AcOH. M.p. 192° (187°). D^{20} 2·983. Very sol. CS_2. Spar. sol. abs. EtOH. Forms add. comps. with amines. Dissociates in sunlight $\longrightarrow I_2 + C_2I_2$.

Datta, Prosad, *J. Am. Chem. Soc.*, 1917, **39**, 451.
Biltz, Küppers, *Ber.*, 1904, **37**, 4415.

Tetraiodoisophthalic Acid



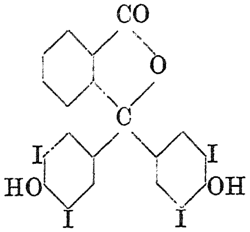
$C_8H_2O_4I_4$ MW, 670

Prisms from AcOH. M.p. 308–12° decomp. Sol. MeOH. Very spar. sol. Et_2O, AcOH.

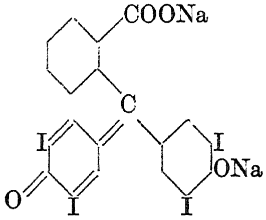
Rupp, *Ber.*, 1896, **29**, 1632.

Tetraiodomethane.
See Carbon tetraiodide.

3′ : 5′ : 3″ : 5″-Tetraiodophenolphthalein
(*Iodophthaleinum, B.P.*)



I



II

$C_{20}H_{10}O_4I_4$ MW, 822
$C_{20}H_8O_4I_4Na_2.3H_2O$ MW, 920

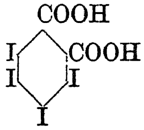
Needles from EtOH–Me_2CO. M.p. 270–1° decomp. Very sol. Py. Sol. alkalis. Spar. sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, AcOEt.

Diacetyl: needles from C_6H_6. M.p. 246°.
Dibenzoyl: plates from C_6H_6. M.p. 298°.

The disodium salt (II) is used as a contrast medium in X-radiography in biliary and gall bladder diseases. It is a bluish-violet, odourless, crystalline powder with an astringent taste. Sol. H_2O, slightly sol. EtOH. Decomp. on heating.

Orndorff, Mahood, *J. Am. Chem. Soc.*, 1918, **40**, 937.

Tetraiodophthalic Acid



$C_8H_2O_4I_4$ MW, 670

Needles from $PhNO_2$. M.p. 324–7°. Very spar. sol. EtOH, Et_2O, AcOH.

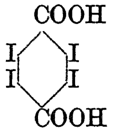
Mono-Me ester: $C_9H_4O_4I_4$. MW, 684. Yellow cryst. from AcOH. M.p. 298° decomp.

Anhydride: $C_8O_3I_4$. MW, 652. Yellow needles from $PhNO_2$. M.p. 325° (320–5°).

Rupp, *Ber.*, 1896, **29**, 1634.
Pratt, Shup, *J. Am. Chem. Soc.*, 1918, **40**, 254.

Tetraiodopyrrole.
See Iodol.

Tetraiodoterephthalic Acid



$C_8H_2O_4I_4$ MW, 670

Prisms from AcOH. M.p. 315–20° decomp. Sol. EtOH. Spar. sol. Et_2O, AcOH, C_6H_6.

Di-Me ester: $C_{10}H_6O_4I_4$. MW, 698. Cryst. from xylene. M.p. 310–12°. Very spar. sol. H_2O.

Di-Et ester: $C_{12}H_{10}O_4I_4$. MW, 726. Cryst. from xylene. M.p. 262·5°.

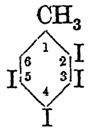
Dipropyl ester: $C_{14}H_{14}O_4I_4$. MW, 754. M.p. 239°.

Dichloride: $C_8O_2Cl_2I_4$. MW, 707. Cryst. from $CHCl_3$. M.p. 279°. Very stable towards NaOH.

Lütjens, *Ber.*, 1896, **29**, 2836.
Rupp, *ibid.*, 1629.

Tetraiodotetramethylmethane.
See Pentaerythrityl tetraiodide.

2 : 3 : 4 : 5-Tetraiodotoluene



$C_7H_4I_4$ MW, 596

Yellow needles from C_6H_6. M.p. 284–5°. Mod. sol. C_6H_6. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 506.

2 : 3 : 4 : 6-Tetraiodotoluene.
Needles from C_6H_6. M.p. 170°. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 133.

2 : 3 : 5 : 6-Tetraiodotoluene.
Needles from C_6H_6. M.p. 125°. Sol. C_6H_6. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 499.

1 : 3 : 4 : 6-Tetraketo-1 : 6-diphenylhexane.
See Diphenacyl Diketone.

Tetralin.
See Tetrahydronaphthalene.

Tetralol.
See Tetrahydronaphthol.

Tetralone.
See Ketotetrahydronaphthalene.

3 : 4 : 3′ : 4′-Tetramethoxydeoxybenzoin.
See Deoxyveratroin.

2 : 3 : 4 : 6-Tetramethylacetanilide.
See under Isoduridine.

Tetramethylacetone.
See Di-isopropyl Ketone.

Tetramethylalloxantin.
See Amalic Acid.

Tetramethylammonium bromide

$(CH_3)_4NBr$

$C_4H_{12}NBr$ MW, 154

Volatile cryst. Sublimes with decomp. above 230°. Sol. H_2O. Spar. sol. abs. EtOH. Insol. Et_2O, $CHCl_3$.

Duvillier, Buisine, *Ann. chim. phys.*, 1881, **23**, 327.
Schmidt, *Ann.*, 1892, **267**, 265.

Tetramethylammonium chloride

$(CH_3)_4NCl$

$C_4H_{12}NCl$ MW, 109·5

Volatile cryst. Decomp. above 230° to trimethylamine + CH_3Cl. Sol. H_2O, EtOH. Insol. $CHCl_3$.

Vincent, Chappuis, *Bull. soc. chim.*, 1886, **45**, 502.

Tetramethylammonium hydroxide

$(CH_3)_4N{\cdot}OH$

$C_4H_{13}ON$ MW, 91

Pentahydrate (+ $5H_2O$): hygroscopic needles. M.p. 63°. Decomp. on dist. to trimethylamine and CH_3OH. Very sol. H_2O. Absorbs CO_2 rapidly. Gentle warming at 40–50° ⟶ *trihydrate* (+ $3H_2O$), m.p. 60°. Careful warming *in vacuo* ⟶ *monohydrate* (+ $1H_2O$), decomp. at 130° to trimethylamine and CH_3OH. Very powerful alkali, comparable with NaOH. The salts form a large number of add. comps. with metal and other halides.

Walker, Johnston, *J. Chem. Soc.*, 1905, **87**, 958.
Schmidt, *Ann.*, 1892, **267**, 267.
Hofmann, *Ann.*, 1851, **79**, 18.

Tetramethylammonium iodide

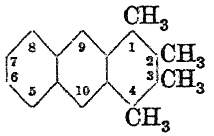
$(CH_3)_4NI$

$C_4H_{12}NI$ MW, 201

Prisms. Decomp. above 230°. Spar. sol. cold H_2O, abs. EtOH. Insol. Et_2O, $CHCl_3$. Triboluminescent.

Chablay, *Ann. chim.*, 1914, **1**, 477.
Hofmann, *Ann.*, 1851, **79**, 16.

1 : 2 : 3 : 4-Tetramethylanthracene



$C_{18}H_{18}$ MW, 234

Yellow cryst. M.p. 135·5–136·5°.
Picrate: m.p. 165–6°.

Hewett, *J. Chem. Soc.*, 1940, 293.

1 : 2 : 5 : 6-Tetramethylanthracene.
Pale yellow cryst. from AcOH. M.p. 204°.
Picrate: m.p. 187–9°.

Nichol, Sandin, *J. Am. Chem. Soc.*, 1947, **69**, 2256.

1 : 2 : 7 : 8-Tetramethylanthracene.
Red needles from C_6H_6–EtOH. M.p. 183–5°.

Nichol, Sandin, *J. Am. Chem. Soc.*, 1947, **69**, 2256.

1 : 2 : 9 : 10-Tetramethylanthracene.
Yellow cryst. M.p. 52–4°.
Trinitrobenzene add. comp.: purple cryst. M.p. 170·5—171·5°.

Sandin, Kitchen, Fieser, *J. Am. Chem. Soc.*, 1943, **65**, 2018.

1 : 3 : 5 : 7-Tetramethylanthracene. Yellowish leaflets from EtOH. M.p. 163–4°. *Picrate*: m.p. 189–90°.

Seer, *Monatsh.*, 1912, **33**, 42.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

1 : 3 : 6 : 8-Tetramethylanthracene. Pale yellow plates with green fluor. from AcOH. M.p. 281–3° (280°). Sol. hot EtOH, hot Et_2O. Mod. sol. $CHCl_3$. CrO_3 in AcOH ⟶ 1 : 3 : 6 : 8-tetramethylanthraquinone.

Seer, *Monatsh.*, 1912, **33**, 36.
Anschütz, *Ann.*, 1886, **235**, 174.
Dewar, Jones, *J. Chem. Soc.*, 1904, **85**, 217.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

1 : 3 : 9 : 10-Tetramethylanthracene. Greenish-yellow plates. M.p. 85–7°. *Picrate*: m.p. 136·5–138°.

Fieser, Heymann, *J. Am. Chem. Soc.*, 1942, **64**, 376.

1 : 4 : 5 : 8-Tetramethylanthracene. M.p. 270°.

Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

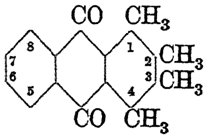
2 : 3 : 6 : 7-Tetramethylanthracene. Pale yellow plates from CS_2. M.p. 301°. Sublimes. Sol. AcOH, CS_2, C_6H_6. Spar. sol. EtOH.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2331.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

2 : 3 : 9 : 10-Tetramethylanthracene. Yellow plates. M.p. 138–40°.

Sandin, Kitchen, *J. Am. Chem. Soc.*, 1945, **67**, 1305.

1 : 2 : 3 : 4-Tetramethylanthraquinone



$C_{18}H_{16}O_2$ MW, 264

Yellow needles from AcOH. M.p. 232–3°.

Hewett, *J. Chem. Soc.*, 1940, 293.

1 : 2 : 5 : 6-Tetramethylanthraquinone. Yellowish-brown needles from AcOH. M.p. 201–2°.

Nichol, Sandin, *J. Am. Chem. Soc.*, 1947, **69**, 2256.

1 : 3 : 5 : 7-Tetramethylanthraquinone. Yellow needles from EtOH. M.p. 235°. Sol. AcOH, C_6H_6. Very spar. sol. EtOH. Sol. conc. H_2SO_4 with deep red col. Sublimes. HNO_3 (D 1·1) at 190–210° ⟶ anthraquinone-1 : 3 : 5 : 7-tetracarboxylic acid. Dist. with Zn dust ⟶ 1 : 3 : 5 : 7-tetramethylanthracene.

Seer, *Monatsh.*, 1912, **33**, 39.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

1 : 3 : 6 : 7-Tetramethylanthraquinone. Yellow needles. M.p. 196°.

Fieser, Fieser, *J. Am. Chem. Soc.*, 1935, **57**, 1679.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

1 : 3 : 6 : 8-Tetramethylanthraquinone. M.p. 228–30° (172–3°).

Seer, *Monatsh.*, 1912, **33**, 36.
Dewar, Jones, *J. Chem. Soc.*, 1904, **85**, 218.
Börnstein, Schliewiensky, Szczesny-Heyl, *Ber.*, 1926, **59**, 2815.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

1 : 4 : 5 : 8-Tetramethylanthraquinone. M.p. 258–60° (235°).

Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

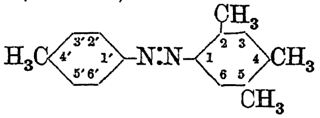
1 : 4 : 6 : 7-Tetramethylanthraquinone. Yellow needles. M.p. 178°.

Fieser, Fieser, *J. Am. Chem. Soc.*, 1935, **57**, 1679.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.
Niederl, Nagel, *J. Am. Chem. Soc.*, 1941, **63**, 307.

2 : 3 : 6 : 7-Tetramethylanthraquinone. Yellow needles or plates from AcOH. M.p. 330°. Sublimes. Sol. conc. H_2SO_4 with deep red col.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2329.
Ellison, Hey, *J. Chem. Soc.*, 1938, 1847.

2 : 4 : 5 : 4′-Tetramethylazobenzene (5-p-*Tolueneazo-ψ-cumene*)



$C_{16}H_{18}N_2$ MW, 238

Yellow needles. M.p. 58°.

Michaelis, Petow, *Ber.*, 1898, **31**, 994.

2:3:2′:3′-Tetramethylazobenzene (3:3′-*Azo-o-xylene*). Orange needles. M.p. 110–11°. Sol. EtOH, Et_2O, C_6H_6.

Noelting, Stricker, *Ber.*, 1888, **21**, 3139.

2:4:2′:4′-Tetramethylazobenzene (4:4′-*Azo-m-xylene*). Red needles from EtOH. M.p. 129° (126°). Sol. EtOH, Et_2O.

Noelting, Stricker, *Ber.*, 1888, **21**, 3141.
Vorländer, Meyer, *Ann.*, 1902, **320**, 128.

2:4:3′:5′-Tetramethylazobenzene (4:5′-*Azo-m-xylene*). Red plates or needles from EtOH. M.p. 46–7°. Sol. common org. solvents.

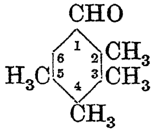
Zincke, Jaenke, *Ber.*, 1888, **21**, 543.

2:5:2′:5′-Tetramethylazobenzene (2:2′-*Azo*-p-*xylene*).
Yellow needles. M.p. 119°.
Noelting, Stricker, *Ber.*, 1888, **21**, 3143.

3:4:3′:4′-Tetramethylazobenzene (4:4′-*Azo*-o-*xylene*).
Red needles. M.p. 140–1°.
Noelting, Stricker, *Ber.*, 1888, **21**, 3140.

3:5:3′:5′-Tetramethylazobenzene (5:5′-*Azo*-m-*xylene*).
Orange needles. M.p. 136–7°. Sol. EtOH, Et_2O.
Noelting, Stricker, *Ber.*, 1888, **21**, 3142.

2 : 3 : 4 : 5-Tetramethylbenzaldehyde



$C_{11}H_{14}O$ MW, 162

Cryst. M.p. 29–30°. B.p. 142–4°/15 mm. Very sol. most org. solvents. Oxidises in air. $KMnO_4$ ⟶ 2 : 3 : 4 : 5-tetramethylbenzoic acid.
Semicarbazone: needles from EtOH. M.p. 221–2°.
Oxime: m.p. 152·5–153·5°.
Auwers, Köckritz, *Ann.*, 1907, **352**, 316.

2 : 3 : 4 : 6-Tetramethylbenzaldehyde.
F.p. 15°. B.p. 136°/10 mm.
Semicarbazone: m.p. 183–5° followed by solidification and remelting at 218–21° decomp.
Oxime: m.p. 136–7°.
Smith, Nichols, *J. Org. Chem.*, 1941, **6**, 489.

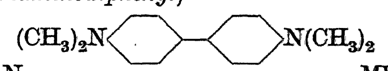
2 : 3 : 5 : 6-Tetramethylbenzaldehyde.
F.p. 20°. B.p. 135°/11 mm. n_D^{20} 1·5560.
Semicarbazone: m.p. 268–70° decomp.
Oxime: m.p. 124·5–125·5°.
Smith, Nichols, *J. Org. Chem.*, 1941, **6**, 489.
Fuson *et al.*, *J. Am. Chem. Soc.*, 1944, **66**, 1109.

1 : 2 : 3 : 4-Tetramethylbenzene.
See Prehnitene.

1 : 2 : 3 : 5-Tetramethylbenzene.
See Isodurene.

1 : 2 : 4 : 5-Tetramethylbenzene.
See Durene.

***N* : *N*′-Tetramethylbenzidine** (4 : 4′-*Tetramethyldiaminodiphenyl*)

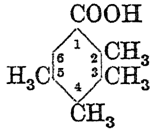


$C_{16}H_{20}N_2$ MW, 240

Needles from C_6H_6–pet. ether. M.p. 198° (193°). B.p. above 360°. Very sol. $CHCl_3$. Sol. hot AcOEt, hot C_6H_6. Spar. sol. Et_2O, boiling ligroin. Very spar. sol. MeOH, EtOH. Non-volatile in steam.

Monomethochloride: m.p. 228°. Sol. H_2O, EtOH.
Monomethiodide: needles from H_2O. M.p. 263°.
König, Seifert, *Ber.*, 1934, **67**, 2119.
Willstätter, Kalb, *Ber.*, 1904, **37**, 3765.
Ullmann, Dieterle, *ibid.*, 29.

2 : 3 : 4 : 5-Tetramethylbenzoic Acid (*Prehnitene-carboxylic acid*)



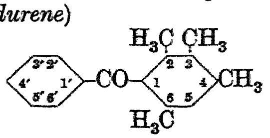
$C_{11}H_{14}O_2$ MW, 178

Needles from EtOH or ligroin. M.p. 168–9° (165°). Volatile in steam.
Me ester: $C_{12}H_{16}O_2$. MW, 192. Needles from $CHCl_3$. M.p. 36°. Sol. EtOH, Et_2O, $CHCl_3$, ligroin.
Amide: $C_{11}H_{15}ON$. MW, 177. Needles from EtOH. M.p. 222°. Spar. sol. Et_2O.
v. Meyer, Molz, *Ber.*, 1897, **30**, 1279.
Smith, Harris, *J. Am. Chem. Soc.*, 1935, **57**, 1292.

2:3:4:6-Tetramethylbenzoic Acid (*Isodurene-carboxylic acid*).
Prisms from ligroin. M.p. 164–5°.
Chloride: b.p. 102–3°/3 mm. D_{20}^{20} 1·1087. n_D^{20} 1·538.
Amide: needles from H_2O. M.p. 141–2°.
Nitrile: $C_{11}H_{13}N$. MW, 159. Cryst. from EtOH. M.p. 68–9°. Stable to HCl at 200°. HCl at 250° ⟶ isodurene.
Gattermann, *Ber.*, 1899, **32**, 1118.

2 : 3 : 5 : 6 - Tetramethylbenzoic Acid (*Durene-carboxylic acid*).
Prisms from H_2O or ligroin. M.p. 179° (176·5°). Sol. EtOH, boiling H_2O. Volatile in steam. Sublimes. HCl at 200–20° ⟶ durene.
Me ester: leaflets from EtOH. M.p. 59°.
p-*Tolyl ester*: m.p. 138°.
Chloride: m.p. 59–60°. B.p. 105°/6 mm.
Amide: cryst. from H_2O. M.p. 178°.
Nitrile: needles from EtOH. M.p. 76–7°. Conc. HCl at 210–20° ⟶ durene.
Jacobsen, *Ber.*, 1889, **22**, 1223.
Gattermann, *Ber.*, 1899, **32**, 1119.
Meyer, Wöhler, *Ber.*, 1896, **29**, 2572.

2 : 3 : 4 : 6 - Tetramethylbenzophenone (*Benzoylisodurene*)



$C_{17}H_{18}O$ MW, 238

Cryst. M.p. 62–3°. B.p. about 300°.
Essner, Gossin, *Bull. soc. chim.*, 1884, **42**, 171.

2 : 3 : 5 : 6 - Tetramethylbenzophenone (*Benzoyldurene*).

M.p. 119°. B.p. 343–343·5°/725 mm. Very sol. hot EtOH. KOH fusion ⟶ durene + benzoic acid.

Friedel, Crafts, Ador, *Ann. chim. phys.*, 1884, **1**, 511.

2 : 4 : 5 : 4′-Tetramethylbenzophenone.

Viscous oil. B.p. 220°/22 mm.

Oxime: cryst. from EtOH. M.p. 151°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2554.

2 : 4 : 6 : 2′-Tetramethylbenzophenone.

M.p. 111°.

Coops *et al.*, *Rec. trav. chim.*, 1940, **59**, 1109.

2 : 4 : 2′ : 4′-Tetramethylbenzophenone.

B.p. 190°/10 mm., 188°/7 mm. D^{15} 1·043. n_D^{12} 1·5876. Does not react with hydroxylamine or phenylhydrazine. Zn dust in alc. alk. sol. ⟶ 2 : 4-dimethylbenzoic acid.

2 : 4-*Dinitrophenylhydrazone*: m.p. 198°.

Böeseken, *Rec. trav. chim.*, 1907, **26**, 285.
Cohen, *Rec. trav. chim.*, 1919, **38**, 119.

2 : 5 : 2′ : 5′-Tetramethylbenzophenone.

Viscous liq. B.p. 325–7°. Prolonged boiling ⟶ 1 : 4 : 6-trimethylanthracene.

Elbs, *J. prakt. Chem.*, 1887, **35**, 481.

2 : 6 : 2′ : 6′-Tetramethylbenzophenone.

M.p. 134°.

Coops *et al.*, *Rec. trav. chim.*, 1940, **59**, 1109.

3 : 4 : 3′ : 4′-Tetramethylbenzophenone.

Cryst. from EtOH. M.p. 140°. Sol. $CHCl_3$, C_6H_6. Spar. sol. Et_2O.

Oxime: cryst. from EtOH. M.p. 147°.

Phenylhydrazone: yellow micro-cryst. M.p. 130°.

Bistrzycki, Reintke, *Ber.*, 1905, **38**, 844.

3 : 5 : 3′ : 5′-Tetramethylbenzophenone.

M.p. 110°.

2 : 4-*Dinitrophenylhydrazone*: m.p. 239°.

Coops *et al.*, *Rec. trav. chim.*, 1940, **59**, 1109.

2 : 3 : 5 : 6-Tetramethylbenzoquinone.

See Duroquinone.

2 : 2 : 3 : 3-Tetramethylbutane (*Hexamethylethane, di*-tert.-*butyl*)

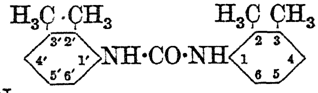
$(CH_3)_3C{\cdot}C(CH_3)_3$

C_8H_{18} MW, 114

Cryst. from Et_2O. M.p. 104° (100·7–101·4°). B.p. 106–7°. Volatile.

Flood, Calingaert, *J. Am. Chem. Soc.*, 1934, **56**, 1211.
Whitmore, Stehman, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3807.

2 : 3 : 2′ : 3′-Tetramethylcarbanilide (2 : 3 : 2′ : 3′-*Tetramethyldiphenylurea*)



$C_{17}H_{20}ON_2$ MW, 268

Cryst. from EtOH. M.p. 242°.

Mazourewitch, *Bull. soc. chim.*, 1924, **35**, 1185.

2 : 4 : 2′ : 4′-Tetramethylcarbanilide (2 : 4 : 2′ : 4′-*Tetramethyldiphenylurea*).

Needles from AcOH. M.p. 263–5°.

Thomson, Wilson, *J. Chem. Soc.*, 1933, 1263.

2 : 5 : 2′ : 5′-Tetramethylcarbanilide (2 : 5 : 2′ : 5′-*Tetramethyldiphenylurea*).

Needles from AcOH. M.p. 285° (sealed tube).

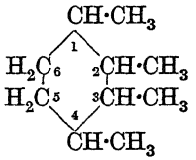
Thomson, Wilson, *J. Chem. Soc.*, 1933, 1263.

3 : 4 : 3′ : 4′-Tetramethylcarbanilide (3 : 4 : 3′ : 4′-*Tetramethyldiphenylurea*).

M.p. 236°.

Mailhe, *Compt. rend.*, 1923, **176**, 903.

1 : 2 : 3 : 4-Tetramethylcyclohexane (*Hexahydroprehnitene*)



$C_{10}H_{20}$ MW, 140

B.p. 84°/5 mm. D_4^{20} 0·8219. n_D^{20} 1·4531.

Mitchell, Marvel, *J. Am. Chem. Soc.*, 1933, **55**, 4278.

1 : 2 : 3 : 5-Tetramethylcyclohexane (*Hexahydroisodurene*).

Cis:

B.p. 168–70°/762 mm. D_4^{20} 0·8166. n_{He}^{20} 1·44847.

Trans:

B.p. 162–4°/765 mm. D_4^{20} 0·8140. n_{He}^{20} 1·44657.

Eisenlohr, *Chem. Abstracts*, 1926, **20**, 171.

1 : 2 : 4 : 5-Tetramethylcyclohexane (*Hexahydrodurene*).

The following compounds have been described:

(i) B.p. 172–4°/730 mm. D_4^{20} 0·7759. n_D^{20} 1·4205.

Pictet, Ramseyer, Kaiser, *Ann. chim.*, 1918, **10**, 297.

(ii) B.p. 169–170·5°/711 mm. D_4^{20} 0·811. n_D^{20} 1·4451.

Willstätter, Hatt, *Ber.*, 1912, **45**, 1473.

(iii) B.p. 160·5–161·5°. $D_4^{13·1}$ 0·7910. $n_D^{13·1}$ 1·437.

Auwers, *Ann.*, 1920, **420**, 108.
D'yakova *et al.*, *J. Gen. Chem. U.S.S.R.*, 1937, 7, 722, (*Chem. Abstracts*, 1937, 31, 5771).

(iv)
Cis :
B.p. 171°/755 mm. D_4^{20} 0·8122. n_{He}^{20} 1·44647.
Trans :
B.p. 166–8°. D_4^{20} 1·8100. n_{He}^{20} 1·44446.

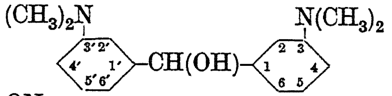
Eisenlohr, *Chem. Abstracts*, 1926, **20**, 171.

1 : 2 : 2 : 3-Tetramethylcyclopentane-1-carboxylic Acid.
See Campholic Acid.

1 : 2 : 3 : 3-Tetramethylcyclopentene.
See Campholene.

Tetramethyldiaminobenzaldehyde.
See under 2 : 4-Diaminobenzaldehyde.

3 : 3′-Tetramethyldiaminobenzhydrol



$C_{17}H_{22}ON_2$ MW, 270

Prisms from Et_2O. M.p. 72–3°. Sols. in acids are colourless.

Baeyer, *Ann.*, 1907, **354**, 194.

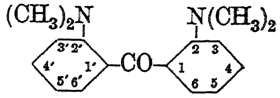
3 : 4′-Tetramethyldiaminobenzhydrol.
Needles from EtOH. M.p. 100–1°. Sol. hot AcOH with yellowish-green col.

Baeyer, *Ann.*, 1907, **354**, 191.

4 : 4′ - Tetramethyldiaminobenzhydrol (*Michler's Hydrol*).
Green leaflets from C_6H_6. M.p. 98° (102–3°). Sol. Et_2O, AcOH, C_6H_6, hot EtOH. Insol. H_2O. 1 mol. Br in cold ⟶ *p*-dimethylaminobenzaldehyde + *p*-bromodimethylaniline. Gives blue col. with H·CHO, CH_3·CHO, chloral, Me_2CO, C_6H_5·CHO, and acetophenone. Intermediate for basic dyes.
Me ether : $C_{18}H_{24}ON_2$. MW, 284. Cryst from ligroin. M.p. 71–2°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. ligroin.
Benzyl ether : plates from ligroin. M.p. 102–3°.
Dimethiodide : leaflets from EtOH. M.p. 195°.
B,$C_6H_3(NO_2)_3$-1 : 3 : 5 : black needles. M.p. 75·5°.

Bogert, Ruderman, *J. Am. Chem. Soc.*, 1922, **44**, 2616.
Cohen, *Rec. trav. chim.*, 1919, **38**, 121.
Badische, D.R.P., 27,032.
Bielecki, Koleniew, *Chem. Zentr.*, 1908, II, 877.
Möhlau, Heinze, *Ber.*, 1902, **35**, 360.
National Aniline & Chemical Co., U.S.P., 1,942,820, (*Chem. Zentr.*, 1934, I, 2826).

2 : 2′-Tetramethyldiaminobenzophenone



$C_{17}H_{20}ON_2$ MW, 268

Yellow prisms or needles from EtOH. M.p. 122° (117–18°). Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Volatile in steam. Does not form oxime or phenylhydrazone.
Picrate : yellow leaflets from EtOH. Decomp. at 160–2°.

Baeyer, *Ber.*, 1905, **38**, 2764.
Bertram, *J. prakt. Chem.*, 1902, **65**, 340.

3 : 3′-Tetramethyldiaminobenzophenone.
Yellow prisms from EtOH.Aq. M.p. 59–60°. Sol. most org. solvents. Sols. in acids are colourless.

Baeyer, *Ann.*, 1907, **354**, 193.

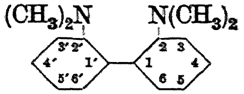
3 : 4′-Tetramethyldiaminobenzophenone.
Brownish-yellow cryst. from EtOH or Et_2O. M.p. 77–78·5°.
Hydrochloride : needles from EtOH. M.p. 278–80° decomp. Decomp. by H_2O.

Baeyer, *Ann.*, 1907, **354**, 190.

4 : 4′-Tetramethyldiaminobenzophenone (*Michler's Ketone*).
Leaflets from EtOH. M.p. 179° (172–172·5°). B.p. above 360° decomp. Sol. warm C_6H_6. Mod. sol. EtOH. Very spar. sol. Et_2O. Insol. H_2O. NaHg in EtOH ⟶ 4 : 4′-tetramethyldiaminobenzhydrol. Intermediate for basic dyes.
Oxime : cryst. from EtOH. M.p. 233°.
Phenylhydrazone : needles from C_6H_6–EtOH. M.p. 174–5°.
Di-Me acetal : pale yellow needles from MeOH. M.p. 130°.
Di-Et acetal : m.p. 118°.
Dimethobromide : yellowish plates + $2H_2O$ from EtOH.Aq. M.p. anhyd. 168°.
Dimethiodide : yellowish leaflets from EtOH. M.p. 105°. At 150° decomp. into its components.
Hydrazone : yellow needles from EtOH. M.p. 150°.
Azine : brownish-red prisms from xylene. M.p. 253°.
Picrate : purple-red prisms. M.p. 156–7°.

Michler, Moro, *Ber.*, 1879, **12**, 1168.
Michler, *Ber.*, 1876, **9**, 716.
Fehrmann, *Ber.*, 1887, **20**, 2845.

2 : 2′-Tetramethyldiaminodiphenyl



$C_{16}H_{20}N_2$ MW, 240

Plates from pet. ether. M.p. 72–3°.
Hydriodide: m.p. 256–7°.
Methiodide: prisms from H_2O. M.p. 190–2°.

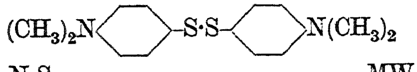
Shaw, Turner, *J. Chem. Soc.*, 1933, 139.

3 : 3′-Tetramethyldiaminodiphenyl.
Needles from EtOH. M.p. 126–8°.

Dutt, *J. Chem. Soc.*, 1926, 1181.

4 : 4′-Tetramethyldiaminodiphenyl.
See N : *N′*-Tetramethylbenzidine.

Tetramethyldiaminodiphenyl disulphide (*Dithiodimethylaniline, tetramethyldithioaniline*)



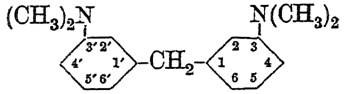
$C_{16}H_{20}N_2S_2$ MW, 304

Yellow needles from EtOH. M.p. 118°. Sol. CS_2. Spar. sol. EtOH, Et_2O, ligroin, hot C_6H_6. Insol. H_2O.

Söderbäck, *Ann.*, 1919, **419**, 276.
Merz, Weith, *Ber.*, 1886, **19**, 1571.

Tetramethyldiaminodiphenyl Ether.
See under N-Dimethyl-*p*-aminophenol.

3 : 3′ - Tetramethyldiaminodiphenyl - methane



$C_{17}H_{22}N_2$ MW, 254

Yellowish oil with green fluor.
Dimethiodide: needles from H_2O. M.p. 165°.

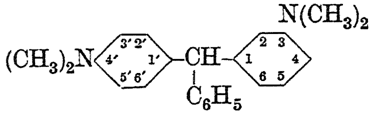
Scholl, *Monatsh.*, 1918, **39**, 236.

4 : 4′ - Tetramethyldiaminodiphenyl - methane (*Methane Base*).

Leaflets or plates from EtOH or ligroin. M.p. 91°. B.p. 390°. Non-volatile in steam. Sol. Et_2O, C_6H_6, hot EtOH. Sol. acids. Ox. ⟶ Michler's Hydrol ⟶ Michler's Ketone.
B,$C_6H_4(NO_2)_2$-1 : 3 : red cryst. M.p. 76°.
B,$C_6H_3(NO_2)_3$-1 : 3 : 5 : violet needles. M.p. 114°.
Monopicrate: yellow leaflets. M.p. 185°.
Dipicrate: m.p. 178°.
Dimethiodide: yellow leaflets. Becomes green at 193° and melts at 214° decomp.
N : N′-*Dioxide*: needles + $2H_2O$ from EtOH–Et_2O. M.p. 147°, anhyd. 156°. Very sol. H_2O, EtOH. Reacts alkaline in solution. *B,2HCl*: m.p. 165·5–166° decomp. *Dipicrate*: yellow needles from EtOH. M.p. 150·5–151° decomp.

Cohn, *Chem.-Ztg.*, 1900, **24**, 564.
Fischl, *Monatsh.*, 1914, **35**, 531.
Votoček, Krauz, *Ber.*, 1909, **42**, 1604.
Nathansohn, Müller, *Ber.*, 1889, 22, 1882.
Höchst, D.R.P., 107,718, (*Chem. Zentr.*, 1900, I, 1110).

3 : 4′ - Tetramethyldiaminotriphenyl - methane



$C_{23}H_{26}N_2$ MW, 330

Cryst. from EtOH. M.p. 83–4°. Mod. sol. Et_2O, hot EtOH.

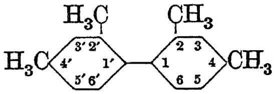
Baeyer, *Ann.*, 1907, **354**, 197.

4 : 4′ - Tetramethyldiaminotriphenyl - methane (*Leuco-Malachite Green*).

Cryst. in three forms. (i) Needles from C_6H_6. M.p. 102°. (ii) Plates from EtOH. M.p. 93–4°. (iii) Cryst. of lower indefinite m.p. Distils undecomp. in small quantities. Sol. Et_2O, C_6H_6, toluene. Mod. sol. EtOH. Spar. sol. ligroin. Insol. H_2O.
B,$C_6H_3(NO_2)_3$-1 : 3 : 5 : m.p. 88·5–89°.
Dimethiodide: plates or needles from H_2O. M.p. 231° (218–22°) decomp. into components.
N : N′-*Dioxide*: needles + 2 or $4H_2O$ from $CHCl_3$. M.p. 131·5–132·5°, anhyd. 188–9°. Very sol. H_2O, EtOH. Very spar. sol. Et_2O, ligroin. Reacts alkaline.

Nencki, *Monatsh.*, 1888, **9**, 1148.
Fischer, *Ann.*, 1881, **206**, 122, 136.
Fischer, Fischer, Lehmann, *Ber.*, 1879, **12**, 798.

2 : 4 : 2′ : 4′-Tetramethyldiphenyl



$C_{16}H_{18}$ MW, 210

Cryst. from EtOH. M.p. 41°. B.p. 288°/722 mm. Sol. Et_2O, C_6H_6, warm EtOH.

Scholl, Liese, Michelson, Grunewald, *Ber.*, 1910, **43**, 513.

2 : 4 : 2′ : 6′-Tetramethyldiphenyl.
B.p. 114–15°/5 mm. n_D^{20} 1·5648.

Carlin, *J. Am. Chem. Soc.*, 1945, **67**, 928.

2 : 5 : 2′ : 5′-Tetramethyldiphenyl.
Cryst. from EtOH. M.p. 50°. B.p. 284°/732 mm. Sol. Et_2O, C_6H_6.

Ullmann, Meyer, *Ann.*, 1904, **332**, 47.

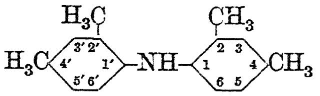
2 : 6 : 2′ : 6′-Tetramethyldiphenyl.
M.p. 66–7°.

Carlin, *J. Am. Chem. Soc.*, 1945, **67**, 928.

3 : 4 : 3′ : 4′-Tetramethyldiphenyl.
Yellowish needles from EtOH. M.p. 76–7°. Sol. $CHCl_3$, Me_2CO, AcOEt, C_6H_6. Volatile in steam.

Crossley, Hampshire, *J. Chem. Soc.*, 1911, **99**, 726.

2 : 4 : 2′ : 4′-Tetramethyldiphenylamine



$C_{16}H_{19}N$ MW, 225

Cryst. M.p. 58–58·5°. B.p. 305–10°.
B,HCl: m.p. 166° decomp.

Müller, *Ber.*, 1887, **20**, 1042.
Bamberger, Brun, *Helv. Chim. Acta*, 1924, **7**, 118.

3 : 4 : 3′ : 4′-Tetramethyldiphenylamine. Viscous oil. B.p. 340–5° decomp. (330–45°). Volatile in steam.

Müller, *Ber.*, 1887, **20**, 1041.

Tetramethyldiphenylurea.
See Tetramethylcarbanilide.
4 : 8 : 12 : 16-Tetramethyleicosane.
See Bixane.
Tetramethylene.
See Cyclobutane.
Tetramethylene bromide.
See 1 : 4-Dibromobutane.
Tetramethylene chloride.
See 1 : 4-Dichlorobutane.
Tetramethylenediamine.
See Putrescine.
Tetramethylene Glycol (1 : 4-*Dihydroxybutane, butandiol*-1 : 4)

$OH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2OH$

$C_4H_{10}O_2$ MW, 90

Viscous liq. Solidifies in freezing mixture to colourless needles, m.p. 16°. B.p. 230°, 120°/10 mm. Sol. H_2O, EtOH. Spar. sol. Et_2O. D^{20} 1·020. Dil. $HNO_3 \longrightarrow$ succinic acid. Bitter taste. Ppd. from aq. sol. by K_2CO_3.

Di-Me ether: $C_6H_{14}O_2$. MW, 118. B.p. 133°. D^{15} 0·8664. n_D^{15} 1·4031.

Me-Et ether: $C_7H_{16}O_2$. MW, 132. B.p. 145–6°/757 mm. D_4^{18} 0·8484. n_D^{18} 1·4012. Sol. EtOH, Et_2O.

Di-Et ether: $C_8H_{18}O_2$. MW, 146. B.p. 155–7°/730 mm., 59–60°/18 mm. D_4^{20} 0·8455. n_D^{20} 1·40610.

Dipropyl ether: $C_{10}H_{22}O_2$. MW, 174. B.p. 94–5°/20 mm. D_4^{20} 0·8409. n_D^{20} 1·41368.

Di-isopropyl ether: b.p. 77–8°/18 mm. D_4^{20} 0·8310. n_D^{20} 1·40954.

Diacetyl: m.p. 12°. B.p. 230°, 124°/20 mm. D^{15} 1·0479. n_D^{15} 1·4251.

Carbonate: m.p. 59°.

Succinate: *monomer*, m.p. 42°. B.p. 95–6°/2 mm. D_4^{60} 1·1732. n_D^{20} 1·4567. *Dimer*: m.p. 121°.

Azelate: *monomer*, m.p. 9°. B.p. 123–4°/2 mm.

Sebacate: *monomer*, m.p. 6°. B.p. 136–8°/2 mm.

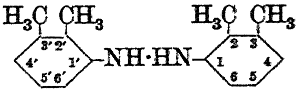
Dibenzoyl: prisms or needles from pet. ether. M.p. 57·5° (53°).

Di-phenylurethane: cryst. from $CHCl_3$. M.p. 183–183·5°.

Di-1-*naphthylurethane*: needles from butyl alcohol. M.p. 198°.

Wojcik, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 2423.
Tallman, *ibid.*, 128.
Müller, Clostermeyer, *Monatsh.*, 1928, **49**, 28.
Bennett, Heathcoat, *J. Chem. Soc.*, 1929, 271.
Böeseken, *Rec. trav. chim.*, 1915, **34**, 100.
I.G., B.Ps 508,548, (*Chem. Abstracts*, 1940, **34**, 779), 508,944, (*Chem. Abstracts*, 1940, **34**, 2866); F.Ps 844,535, (*Chem. Abstracts*, 1940, **34**, 7298), 845,305, (*Chem. Abstracts*, 1941, **35**, 1068).

Tetramethyleneimine.
See Pyrrolidine.
Tetramethylene iodide.
See 1 : 4-Di-iodobutane.
Tetramethylene oxide.
See Tetrahydrofuran.
Tetramethylene sulphide.
See Tetrahydrothiophene.
Tetramethylene sulphone.
See under Tetrahydrothiophene.
Tetramethylethane.
See Dimethylbutane.
Tetramethylethylene.
See 2 : 3-Dimethyl-2-butylene.
Tetramethylethylene Glycol.
See Pinacol.
2 : 6 : 11 : 15-Tetramethylhexadecane.
See Crocetane.
Tetramethylhydracrylic Acid.
See 2-Hydroxy-1 : 1-dimethylisovaleric Acid.
2 : 3 : 2′ : 3′-Tetramethylhydrazobenzene (3 : 3′-*Hydrazo*-o-*xylene*)



$C_{16}H_{20}N_2$ MW, 240

Colourless needles from EtOH. M.p. 149–50° (139–40°). Sol. most org. solvents. Oxidises easily in air.

Bamberger, *Ber.*, 1926, **59**, 428.

2 : 4 : 2′ : 4′-Tetramethylhydrazobenzene (4 : 4′-*Hydrazo*-m-*xylene*).
Needles from EtOH. M.p. 125–6° (120–2°).

Bamberger, *Ber.*, 1926, **59**, 428.

2 : 5 : 2′ : 5′-Tetramethylhydrazobenzene (2 : 2′-*Hydrazo*-p-*xylene*).
Needles from EtOH. M.p. 145°.

Nölting, Stricker, *Ber.*, 1888, **21**, 3143.

3 : 4 : 3′ : 4′-Tetramethylhydrazobenzene (4 : 4′-*Hydrazo*-o-*xylene*).
Needles from EtOH. M.p. 113–14° (106–7°).

Bamberger, *Ber.*, 1926, **59**, 428.

3 : 5 : 3′ : 5′-Tetramethylhydrazobenzene (5 : 5′-*Hydrazo*-m-*xylene*).
Needles from EtOH. M.p. 124–5°. Oxidises readily in air.

Nölting, Stricker, *Ber.*, 1888, **21**, 3142.

Tetramethylhydroquinone.
See Durohydroquinone.

Tetramethylhydurilic Acid.
See Deoxyamalic Acid.

Tetramethylmethane (2-*Methylisobutane, neopentane*, 2 : 2-*dimethylpropane*)

$C(CH_3)_4$

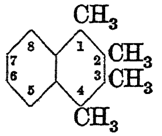
C_5H_{12} MW, 72

F.p. – 19·5°. B.p. 9·4°/760 mm. D_0^0 0·613. n_D^0 1·3513. Forms a mol. complex (from MeOH, m.p. 147–52° decomp.) with deoxycholic acid.

Whitmore, Fleming, *J. Am. Chem. Soc.*, 1933, **55**, 3803.

Tetramethylmethane - tetracarboxylic Acid.
See Methane-tetracetic Acid.

1 : 2 : 3 : 4-Tetramethylnaphthalene



$C_{14}H_{16}$ MW, 184

Needles from EtOH. M.p. 106·5–107·5°.
Picrate : vermilion needles from EtOH. M.p. 182–3°.

Hewett, *J. Chem. Soc.*, 1940, 293.

1 : 2 : 4 : 8-Tetramethylnaphthalene.
Oil. B.p. 150°/10 mm.
Picrate : red needles. M.p. 145·5°.

Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 324.

1 : 2 : 5 : 6-Tetramethylnaphthalene.
Cryst. from EtOH. M.p. 118°. B.p. 150–60°/10 mm.
Picrate: red needles. M.p. 156–7° (154–154·5°).
Styphnate : cryst. from C_6H_6. M.p. 166° (162°).
$C_{14}H_{16},C_6H_3(NO_2)_3$-1 : 3 : 5 : orange-red needles from MeOH. M.p. 180–180·5° (178·5–179°).

Brunner, Hofer, Stein, *Monatsh.*, 1933, **63**, 96.
Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 320.

1 : 2 : 5 : 7-Tetramethylnaphthalene.
B.p. 155–8°/12 mm.
Picrate : orange-yellow needles from MeOH. M.p. 144–5°.
Styphnate : orange-yellow needles from MeOH. M.p. 144–5°.
$C_{14}H_{16},C_6H_3(NO_2)_3$-1 : 3 : 5 : pale yellow needles from EtOH. M.p. 167–8°.

Hosking, Brandt, *Ber.*, 1935, **68**, 289.
Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 323.

1 : 2 : 5 : 8-Tetramethylnaphthalene.
Oil. B.p. 150°/9 mm.
Picrate : red cryst. M.p. 137–8° (146–7°).
$C_{14}H_{16},C_6H_3(NO_2)_3$-1 : 3 : 5 : orange - yellow needles. M.p. 158–9° (166–7°).

Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 322.
Ruzicka, Schinz, Müller, *Helv. Chim. Acta*, 1944, **27**, 195.

1 : 2 : 6 : 8-Tetramethylnaphthalene.
Oil. B.p. 166–8°/15 mm.
Picrate : orange-red needles. M.p. 133·5–134°.
Styphnate : orange needles. M.p. 135–6°.
sym.-*Trinitrobenzene add. comp.*: yellowish-brown needles. M.p. 153–4°.

Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, **16**, 321.
Ruzicka, Schinz, Müller, *Helv. Chim. Acta*, 1944, **27**, 195.

2 : 2 : 4 : 4-Tetramethylpentane

$CH_3{\cdot}C(CH_3)_2{\cdot}CH_2{\cdot}C(CH_3)_2{\cdot}CH_3$

C_9H_{20} MW, 128

M.p. – 66·6°. B.p. 122–3°/760 mm. D_4^{25} 0·71566. n_D^{20} 1·40677.

Whitmore, Southgate, *J. Am. Chem. Soc.*, 1938, **60**, 2571.
Smith, *Journal of Research of the National Bureau of Standards*, 1941, **26**, 129, (*Chem. Abstracts*, 1945, **35**, 2760).

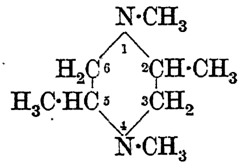
2 : 2 : 4 : 4-Tetramethylpentanol-3.
See Di-*tert*.-butylcarbinol.

2 : 2 : 4 : 4-Tetramethylpentanone-3.
See Pivalone.

Tetramethylphenol.
See Durenol *and* Prehnitenol.

Tetramethylphenylenediamine.
See under Phenylenediamine.

1 : 2 : 4 : 5-Tetramethylpiperazine



$C_8H_{18}N_2$ MW, 142

Syrup.
B,2HI : m.p. 257° decomp.
B,HI : cryst. M.p. 178° decomp.
Dimethiodide : cryst. from MeOH. M.p. 250°.
Dihydroxymethylate : cryst. from Et_2O–pet. ether. M.p. 224°.

Abderhalden, Haas, *Z. physiol. Chem.*, 1925, **149**, 94.

1 : 2 : 4 : 6-Tetramethylpiperazine.
Colourless liq. B.p. 163–4°. Misc. with H_2O and most org. solvents in all proportions.
B_2,H_2PtCl_6 : orange needles + $2H_2O$ from H_2O. Darkens at 270°. Decomp. at 275°.
Monomethiodide : needles from Et_2O–EtOH. M.p. 227°.
d-*Camphor*-β-*sulphonate* : needles or prisms from EtOH–Me_2CO. M.p. 223°. $[\alpha]_{5461}$ + 21·6° in H_2O.
d-α-*Bromocamphor*-β-*sulphonate* : cryst. from Me_2CO. M.p. 175°. $[\alpha]_{5461}$ + 97·4° in H_2O.
d-α-*Bromocamphor*-π-*sulphonate* : needles from Me_2CO. M.p. 249°. $[\alpha]_{5461}$ + 88·1° in H_2O.
Picrate : yellow cryst. Decomp. at 280°.

Pope, Read, *J. Chem. Soc.*, 1914, **105**, 224.

2 : 2 : 5 : 5-Tetramethylpiperazine.
M.p. 85–7°. B.p. 168–71°.
B,2HCl : melts above 350°.
Dinitroso deriv. : cryst. from AcOH. M.p. 208–10°.

Conant, Aston, *J. Am. Chem. Soc.*, 1928, **50**, 2788.
McElvain, Pryde, *J. Am. Chem. Soc.*, 1949, **71**, 326.

2 : 3 : 5 : 6-Tetramethylpiperazine.
There are five optically inactive stereoisomers.

I.
Colourless needles. M.p. 45°. B.p. 177–8°. Readily sol. H_2O, $CHCl_3$.
Dihydrate : needles from Me_2CO. M.p. 84–5°. Very sol. H_2O ⟶ alk. sol.
B,2HCl : prisms from EtOH.Aq. Does not melt below 300°.
Dinitroso deriv. : needles from EtOH. M.p. 157°.
Dibenzoyl : plates from EtOH. M.p. 247–8°.
Di-p-*toluenesulphonyl* : cryst. from Py. M.p. 308–9°.

II.
Liq. B.p. 183°. Very sol. H_2O.
B,2HCl : prisms from EtOH. Does not melt below 300°.
Dinitroso deriv. : yellow prisms from EtOH. M.p. 101–2°.
Dibenzoyl : prisms from Me_2CO–pet. ether. M.p. 175–6°.
Mono-p-*toluenesulphonyl* : needles from EtOH. M.p. 81–2°.
Di-p-*toluenesulphonyl* : plates from Py.Aq. M.p. 222°.
d-*Camphor*-10-*sulphonate* : m.p. 89–90°. $[\alpha]^{15}_{5461}$ + 35·7° in $CHCl_3$.

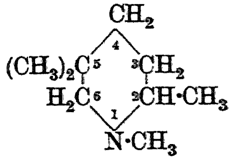
III.
Colourless cryst. M.p. 67–8°. B.p. 195–6°.
B,2HCl : plates + $1H_2O$ from EtOH.Aq. Does not melt below 300°.
Dinitroso deriv. : yellow prisms from EtOH. M.p. 173–4°.
Monobenzoyl : prisms from pet. ether. M.p. 85°.
Dibenzoyl : prisms from EtOH. M.p. 163–4°.
Mono-p-*toluenesulphonyl* : plates from EtOH.Aq., or pet. ether. M.p. 138–9°.
N-*Me* : $C_9H_{20}N_2$. MW, 156. M.p. 4–5°. B.p. 201–2°. Very hygroscopic. *Monohydrate* : prisms from Me_2CO. M.p. 73–4°. *B,HI* : prisms from EtOH or H_2O. M.p. 161–2°. *B,2HCl* : needles from EtOH.Aq.–Me_2CO. M.p. about 300°. *B,2HI* : prisms from EtOH. M.p. 240° decomp. *Nitroso deriv.* : m.p. 24–5°. B.p. 155–7°/15 mm.
N : N-*Di-Me* : $C_{10}H_{22}N_2$. MW, 170. B.p. 211–12°. *Monomethiodide* : needles from MeOH–Me_2CO. M.p. 272–4°.

IV.
Hydrated needles from H_2O. M.p. 53–5°.
B,2HCl : prisms from H_2O. Does not melt below 300°.
Dinitroso deriv. : yellow prisms from Me_2CO. M.p. 189–90°.

V.
B,2HCl : prisms from EtOH.Aq.
Dinitroso deriv. : yellow prisms from EtOH or Me_2CO. M.p. 116–17°.
Dibenzoyl : needles from Me_2CO or pet. ether. M.p. 146–7°.

Stoehr, Brandes, *J. prakt. Chem.*, 1897, **55**, 74.
Kipping, *J. Chem. Soc.*, 1937, 368; 1932, 1336; 1931, 1160; 1929, 2889.

1 : 2 : 5 : 5-Tetramethylpiperidine



$C_9H_{19}N$ MW, 141

Liq. B.p. 147–8°.
B,HCl : m.p. about 221° decomp.

Mannich, Lesse, *Arch. Pharm.*, 1933, **271**, 92, (*Chem. Abstracts*, 1934, **28**, 477).

2 : 2 : 4 : 6-Tetramethylpiperidine.
B.p. 152–3°.
Picrate : m.p. 182–3°.

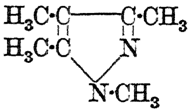
Matter, *Helv. Chim. Acta*, 1948, **31**, 612.

2 : 2 : 6 : 6-Tetramethylpiperidine.
Liq. B.p. 155·5–156·5°. Volatile in steam. $D_4^{16.2}$ 0·8367.
Hydrate: needles. M.p. 28°.
B_2,H_2SO_4: cryst. M.p. 270°.
B,H_2SO_4: prisms from H_2O. M.p. 174°.
B,HNO_2: cryst. from $CHCl_3$. Decomp. at 270°.
$B,HAuCl_4$: yellow needles from H_2O. M.p. 165°.
B_2,H_2PtCl_6: orange-red prisms from H_2O. M.p. 262°.
N-*Benzoyl*: plates from EtOH. M.p. 41–2°.

Franchimont, Friedmann, *Rec. trav. chim.*, 1905, **24**, 404.

2 : 2 : 6 : 6-Tetramethyl-γ-piperidone.
See Triacetonamine.

1 : 3 : 4 : 5-Tetramethylpyrazole

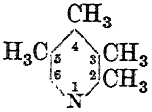


$C_7H_{12}N_2$ MW, 124

Liq. with unpleasant odour. Cryst. on cooling. B.p. 190–3°. Misc. in all proportions with H_2O, Et_2O, EtOH.
Picrate: m.p. 176–8°.

Rojahn, Kühling, *Arch. Pharm.*, 1926, **264**, 337, (*Chem. Abstracts*, 1926, **20**, 2856).
Knorr, Oettinger, *Ann.*, 1894, **279**, 246.

2 : 3 : 4 : 5-Tetramethylpyridine



$C_9H_{13}N$ MW, 135

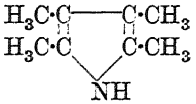
Liq. B.p. 232–4°. Mod. misc. with H_2O. $KMnO_4$ ⟶ pyridine-2 : 3 : 4 : 5-tetracarboxylic acid.
$B,HAuCl_4$: yellow needles from H_2O. M.p. 216–18°.
$B,(HgCl_2)_2,HCl$: needles from H_2O. M.p. 159°.
B_2,H_2PtCl_6: needles from H_2O. M.p. 209–10° decomp.
Picrate: needles from H_2O. M.p. 170–2°.

Ahrens, *Ber.*, 1895, **28**, 796.

2 : 3 : 4 : 6-Tetramethylpyridine.
B.p. 203°/750 mm. D_4^{25} 0·9322. n_D^{25} 1·5087.
Picrate: cryst. from Me_2CO. M.p. 122·5–123°.
Picrolonate: m.p. 225·5° decomp.
Methiodide: m.p. 192–3°.

Nisbet, Pryde, *Nature*, 1951, **168**, 832.

2 : 3 : 4 : 5-Tetramethylpyrrole

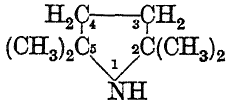


$C_8H_{13}N$ MW, 123

Leaflets from EtOH.Aq. or pet. ether. M.p. 111°. B.p. 130°/7 mm. Misc. with most org. solvents. Volatile in steam with part. decomp.
Picrate: yellow cryst. from C_6H_6, EtOH, or Et_2O. M.p. 130°.
Styphnate: reddish-brown needles. M.p. 159°.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 714.
Nenitzescu, Solomonica, *Ber.*, 1931, **64**, 1928.
Fischer, Zerweck, *Ber.*, 1923, **56**, 525.
Fischer, Bartholomäus, *Z. physiol. Chem.*, 1913, **87**, 269; 1913, **83**, 65.

2 : 2 : 5 : 5-Tetramethylpyrrolidine



$C_8H_{17}N$ MW, 127

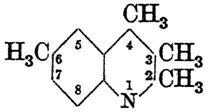
Pale rose-coloured oil. B.p. 108°. With pine splint ⟶ red col. Isatin + H_2SO_4 ⟶ greenish-blue col.
Benzoyl: cryst. M.p. 67·5–68°. Sol. cold pet. ether.

Pace, *Chem. Abstracts*, 1928, **22**, 3890.
Konowalow, Wojnitsch - Sjanoshenski, *Chem. Zentr.*, 1905, II, 830.

2 : 3 : 4 : 5-Tetramethylpyrrolidine.
N-*Et*: $C_{10}H_{21}N$. MW, 155. B.p. 163–5°. D^{15} 0·8064. n_D^{25} 1·4343.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 715.

2 : 3 : 4 : 6-Tetramethylquinoline



$C_{13}H_{15}N$ MW, 185

Cryst. from ligroin or EtOH. M.p. 150°.

Buu-Hoï, Guettier, *Rec. trav. chim.*, 1946, **65**, 502.

2 : 3 : 4 : 7-Tetramethylquinoline.
Cryst. from ligroin or EtOH. M.p. 79°. B.p. 188–90°/28 mm.

Buu-Hoï, Guettier, *Rec. trav. chim.*, 1946, **65**, 502.

2 : 4 : 5 : 7-Tetramethylquinoline.
Cryst. from pet. ether. M.p. 59°.

Mikeska, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 2394.

2 : 4 : 5 : 8-Tetramethylquinoline.
Pale yellow cryst. M.p. 48°. B.p. 168–72°/12 mm. Acid sol. ⟶ bluish-violet fluorescence.
B,HCl: sinters at 243°. M.p. 254°.
Picrate: needles from EtOH. M.p. 161°.

Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 389.

2 : 4 : 6 : 8-Tetramethylquinoline.
Plates from Et_2O or pet. ether. M.p. 86°. B.p. 284–5°. Very sol. Et_2O.
B,H_2SO_4: needles from EtOH–Et_2O. M.p. 243° decomp.

Mikeska, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 2394.

2 : 5 : 6 : 8-Tetramethylquinoline.
Oil. F.p. 20° to cryst. mass. B.p. 297–300°. Very sol. EtOH, Et_2O. Insol. H_2O.

Doebner, Miller, *Ber.*, 1884, **17**, 1710.

Tetramethylsilane.
See Silicon tetramethyl.

Tetramethylsuccinic Acid (2 : 3-*Dimethylbutane*-2 : 3-*dicarboxylic acid*)

$(CH_3)_2C·COOH$
$(CH_3)_2C·COOH$

$C_8H_{14}O_4$ MW, 174

Cryst. M.p. 200°. Sol. 201 parts H_2O at 13·5°. Very sol. EtOH, C_6H_6. Spar. sol. Et_2O, $CHCl_3$, CS_2. Insol. ligroin. $k = 3{\cdot}14 \times 10^{-4}$ at 25°. Heat above m.p. ⟶ anhydride.
Mono-Me ester: $C_9H_{16}O_4$. MW, 188. Prisms from pet. ether. M.p. 68°. $k = 1{\cdot}22 \times 10^{-5}$ at 25°.
Di-Me ester: $C_{10}H_{18}O_4$. MW, 202. Prisms from ligroin. M.p. 31°. Very sol. most org. solvents.
Mono-Et ester: $C_{10}H_{18}O_4$. MW, 202. Viscous oil. Dist. ⟶ anhydride + C_2H_5OH.
Di-Et ester: $C_{12}H_{22}O_4$. MW, 230. B.p. 219°/13 mm. D_4^{20} 0·995. n_{He}^{20} 1·436.
Imide: $C_8H_{13}O_2N$. MW, 155. Needles from C_6H_6–ligroin. M.p. 187°. Spar. sol. ligroin. Sublimes.
p-*Tolylimide*: needles from EtOH.Aq. M.p. 90°.
2-*Naphthylimide*: needles from EtOH.Aq. M.p. 152°.
Anhydride: $C_8H_{12}O_3$. MW, 156. Needles from ligroin. M.p. 147°. B.p. 230·5°. Spar. sol. H_2O, ligroin. Sublimes.
Di-nitrile: $C_8H_{12}N_2$. MW, 136. Plates or prisms from EtOH.Aq. M.p. 169°.
Mono-anilide: cryst. from Et_2O–pet. ether. M.p. 91·5–92°.
Anil: needles from EtOH.Aq. M.p. 88°.
Mono-p-*toluidide*: cryst. M.p. 116–17°.
p-*Tolil*: cryst. M.p. 91·5°.

Auwers, Ungemach, *Ber.*, 1935, **68**, 351.
Auwers, Ottens, *Ber.*, 1924, **57**, 440.
Auwers, Meyer, *Ber.*, 1890, **23**, 297.

Tetramethyltetraethylporphin.
See Aetioporphyrin.

1 : 1 : 2 : 6 - Tetramethyl - 1 : 2 : 3 : 4 - tetrahydronaphthalene.
See Irene.

1 : 1 : 2 : 6-Tetramethyltetralin.
See Irene.

Tetramethylthiourea.
See under Thiourea.

Tetramethylthiuram disulphide

$(CH_3)_2N·CS·S·S·CS·N(CH_3)_2$

$C_6H_{12}N_2S_4$ MW, 240

White cryst. from $CHCl_3$–EtOH. M.p. 146°. Very sol. $CHCl_3$. Spar. sol. EtOH, Et_2O. KCN ⟶ tetramethylthiuram sulphide. Used as accelerator in vulcanisation of rubber.

Cummings, Simmons, *Ind. Eng. Chem.*, 1928, **20**, 1173, (*Brit. Chem. Abstracts*, 1929, B, 28).
Romani, *Chem. Abstracts*, 1922, **16**, 854.
v. Braun, Stechele, *Ber.*, 1903, **36**, 2280.
v. Braun, *Ber.*, 1902, **35**, 820.

Tetramethylthiuram sulphide

$(CH_3)_2N·CS·S·CS·N(CH_3)_2$

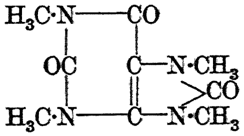
$C_6H_{12}N_2S_3$ MW, 208

Yellow cryst. from EtOH. M.p. 104°. Very sol. EtOH, $CHCl_3$. Spar. sol. cold Et_2O. Stable to dil. acids. Alkalis ⟶ dimethylamine.

Cummings, Simmons, *Ind. Eng. Chem.*, 1928, **20**, 1173, (*Brit. Chem. Abstracts*, 1929, B, 28).
Romani, *Chem. Abstracts*, 1922, **16**, 854.
v. Braun, Stechele, *Ber.*, 1903, **36**, 2280.
v. Braun, *Ber.*, 1902, **35**, 820.

Tetramethylurea.
See under Urea.

Tetramethyluric Acid



$C_9H_{12}N_4O_3$ MW, 224

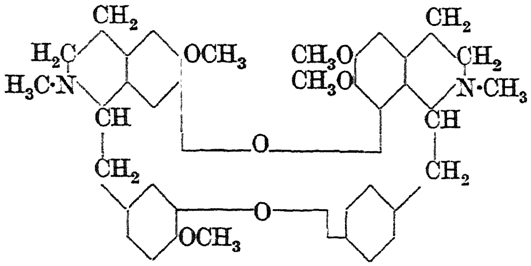
Needles from H_2O. M.p. 228°. Dist. without decomp. Very sol. hot H_2O, boiling $CHCl_3$. Mod. sol. EtOH. Spar. sol. Et_2O, cold H_2O. Decomp. by warm alkalis. Gives murexide reaction. Bitter taste.

Fischer, *Ber.*, 1899, **32**, 2732, 2742; 1884, **17**, 1784.

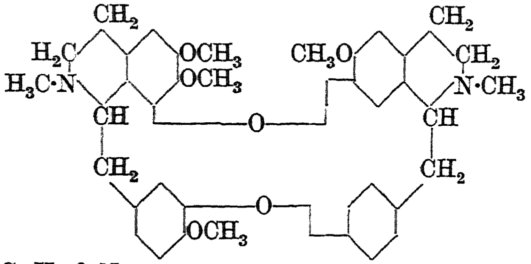
Tetraperinaphthylenecyclo-octadiene.
See Fluorocyclene.

Tetrandrine

Constitution represented by



or



$C_{38}H_{42}O_6N_2$ MW, 622

dl-.

Colourless prisms from MeOH. M.p. 252°.

l-.

See Phaeanthine.

d-.

Alkaloid extracted from roots of *Stephania tetrandra*, S. Moore. Constituent of the Chinese drug, Han-Fang-Chi. Colourless needles. M.p. 217–18°. Sol. Et_2O, Me_2CO, $CHCl_3$. Mod. sol. EtOH. Insol. H_2O, pet. ether. $[\alpha]_D^{26}$ + 252·4° in $CHCl_3$. Poisonous. Affects central nervous system, respiratory and skeletal muscles. Has action on paramecia similar to that of quinine.

B,2HCl : prisms from EtOH. Softens at 263°. Decomp. at 266°. $[\alpha]_D^{27}$ + 224·2° in H_2O.

B,2HBr : needles from EtOH. Decomp. at 270°. $[\alpha]_D^{27}$ + 200·7° in H_2O.

B,2HNO$_3$: prisms from EtOH.Aq. Softens at 205°. Decomp. at 208°. $[\alpha]_D^{26}$ + 211·2° in H_2O.

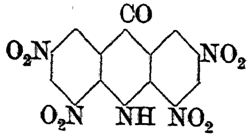
B,(COOH)$_2$: needles. Softens at 147·5–148·5°. Decomp. at 165–70°.

B,2CH$_3$I : needles + $2H_2O$ from Me_2CO.Aq. M.p. 269° decomp.

Picrate : decomp. at 235–42°.

Kondo, Keimatsu, *Ber.*, 1935, **68**, 1505.
Chen, Chen, *J. Biol. Chem.*, 1935, **109**, 681.

1 : 3 : 7 : 9-Tetranitroacridone



$C_{13}H_5O_9N_5$ MW, 375

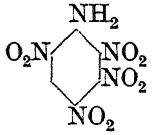
Yellow cryst. Does not melt below 350°. Sol. hot alkalis. Spar. sol. org. solvents. Sublimes. KOH ⟶ cherry-red sol. from which K salt crystallises in red plates.

Lehmstedt, *Ber.*, 1931, **64**, 2383.
Edinger, Arnold, *J. prakt. Chem.*, 1901, **64**, 488.

2 : 4 : 5 : 7-Tetranitro-aloe-emodin.

See Aloetic Acid.

2 : 3 : 4 : 6-Tetranitroaniline



$C_6H_3O_8N_5$ MW, 273

Yellow cryst. from AcOH. M.p. 220°. Slightly hygroscopic. Sol. AcOH, $PhNO_2$. Spar. sol. $CHCl_3$, C_6H_6, ligroin. Insol. cold H_2O. Sol. 6 parts boiling Me_2CO, 3 parts *o*-nitrotoluene at 140°, 3 parts nitroxylene at 150°, and 24 parts boiling xylene. Slowly decomp. with steam at 100°. Warm moist Me_2CO, boiling with H_2O, or with AcONa in Me_2CO.Aq. ⟶ 2 : 4 : 6-trinitro-3-aminophenol. Boiling EtOH ⟶ 2 : 4 : 6-trinitro-3-aminophenetole. Explosive.

N-*Acetyl* : 2 : 3 : 4 : 6-tetranitroacetanilide. M.p. about 170° decomp.

N-*Me* : m.p. 127°.

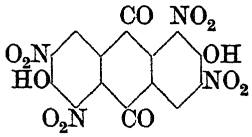
N-*Di-Me* : m.p. 153°.

Forster, Coulson, *J. Chem. Soc.*, 1922, **121**, 1992.
Duin, *Rec. trav. chim.*, 1917, **37**, 115.

Tetranitroanisole.

See under Tetranitrophenol.

1 : 3 : 5 : 7-Tetranitroanthraflavic Acid (1 : 3 : 5 : 7-*Tetranitro*-2 : 6-*dihydroxyanthraquinone*)

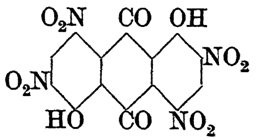


$C_{14}H_4O_{12}N_4$ MW, 420

Yellow needles. Explodes at 307·6° without melting. Very sol. hot H_2O ⟶ red sol. Sol. EtOH, Et_2O ⟶ red sols. Gives salt with NH_3. Boiling conc. HNO_3 ⟶ 2 : 4 : 6-trinitro-*m*-hydroxybenzoic acid.

Wolffenstein, Paar, *Ber.*, 1913, **46**, 596.
Schunck, Roemer, *Ber.*, 1878, **11**, 1178.
Schardinger, *Ber.*, 1875, **8**, 1487.

2:4:6:8-Tetranitroanthrarufin (2:4:6:8-*Tetranitro*-1 : 5-*dihydroxyanthraquinone*)

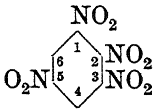


$C_{14}H_4O_{12}N_4$ MW, 420

Yellow plates from conc. HNO_3. Gives cryst. Na, K and Mg hydrated salts. Red. ⟶ 2 : 4 : 6 : 8-tetra-aminoanthrarufin. Boiling conc. HNO_3 ⟶ 2 : 4 : 6-trinitro-*m*-hydroxybenzoic acid.

Wolffenstein, Paar, *Ber.*, 1913, **46**, 592.
Liebermann, *Ber.*, 1879, **12**, 188.

1 : 2 : 3 : 5-Tetranitrobenzene



$C_6H_2O_8N_4$ MW, 258

Yellow cryst. from $CHCl_3$. M.p. 126°. Mod. sol. warm $CHCl_3$, C_6H_6. Spar. sol. Et_2O, CS_2. Aq. alkalis ⟶ picric acid.

Naphthalene add. comp.: orange-yellow cryst. M.p. 139·5°.

Anthracene add. comp.: deep red cryst. M.p. 171°.

Phenanthrene add. comp.: orange-red cryst. M.p. 125°.

α-*Naphthol add. comp.*: brownish-red cryst. M.p. 137°.

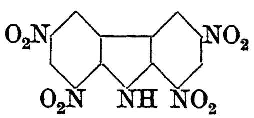
β-*Naphthol add. comp.*: brownish-red cryst. M.p. 130·5°.

Hollemann, *Rec. trav. chim.*, 1930, **49**, 117.
Borsche, *Ber.*, 1923, **56**, 1942.

1 : 2 : 4 : 5-Tetranitrobenzene.
Pale yellow needles from EtOH.Aq. M.p. 188°.

Borsche, Feske, *Ber.*, 1926, **59**, 820.

1 : 3 : 6 : 8-Tetranitrocarbazole



$C_{12}H_5O_8N_5$ MW, 347

Pale yellow plates + $PhNO_2$ from $PhNO_2$, golden-yellow needles from AcOH. Darkens at 200°, m.p. 289°.

N-*Me*: $C_{13}H_7O_8N_5$. MW, 361. Yellow needles from Me_2CO. M.p. 277°. Insol. common org. solvents.

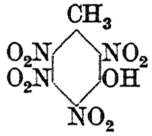
N-*Et*: $C_{14}H_9O_8N_5$. MW, 375. Cryst. from Me_2CO. M.p. 216°.

N-*Phenyl*: $C_{18}H_9O_8N_5$. MW, 423. Cryst. M.p. 244° (255°). Sol. Me_2CO, C_6H_6. Insol. H_2O, EtOH, Et_2O, $CHCl_3$, CCl_4, CS_2.

van Alphen, *Rec. trav. chim.*, 1932, **51**, 183.
Raudnitz, *Ber.*, 1927, **60**, 741.
Borsche, Scholten, *Ber.*, 1917, **50**, 608.

2 : 4 : 5 : 7-Tetranitrochrysazin.
See Chrysamminic Acid.

2 : 4 : 5 : 6-Tetranitro-*m*-cresol



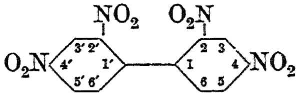
$C_7H_4O_9N_4$ MW, 288

Cryst. from $CHCl_3$. M.p. 175°. Very sol. H_2O, EtOH ⟶ yellow sols. Boiling H_2O ⟶ 2 : 4 : 6-trinitro-3 : 5-dihydroxytoluene. Bitter taste. Explosive.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 34.

Tetranitrodihydroxyanthraquinone.
See Tetranitroanthrarufin *and* Tetranitroanthraflavic Acid.

2 : 4 : 2′ : 4′-Tetranitrodiphenyl



$C_{12}H_6O_8N_4$ MW, 334

Yellow prisms from C_6H_6. M.p. 165–6°. Very sol. AcOH, C_6H_6. Spar. sol. EtOH, Et_2O.

Swann Research Inc., U.S.P., 1,870,627, (*Chem. Zentr.*, 1932, II, 2729).
Ullmann, Bielecki, *Ber.*, 1901, **34**, 2177.

2 : 6 : 2′ : 6′-Tetranitrodiphenyl.
Yellowish needles from AcOH. M.p. 217–18°.

Borsche, Rantscheff, *Ann.*, 1911, **379**, 176.

3 : 4 : 2′ : 4′-Tetranitrodiphenyl.
Pale yellow cubes from MeOH. M.p. 173°.

Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3006.

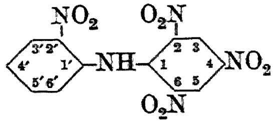
3 : 4 : 3′ : 4′-Tetranitrodiphenyl.
Yellow prisms. M.p. 186° (203–4°). Very sol. AcOH, C_6H_6. Insol. ligroin.

Ullmann, Bielecki, *Ber.*, 1901, **34**, 2179.
Case, *J. Am. Chem. Soc.*, 1942, **64**, 1848.

3 : 5 : 3′ : 5′-Tetranitrodiphenyl.
M.p. 228–9°.

Case, *J. Am. Chem. Soc.*, 1942, **64**, 1848.

2 : 4 : 6 : 2′-Tetranitrodiphenylamine



$C_{12}H_7O_8N_5$ MW, 349

Yellow prismatic needles from AcOH. M.p. 234°. Sol. 600 parts toluene at 20°, 770 parts AcOH at 20°. Sol. hot dil. Na_2CO_3 ⟶ orange-red sol.

Juillard, *Bull. soc. chim.*, 1905, **33**, 1187.

2 : 4 : 6 : 3′-Tetranitrodiphenylamine.
Orange-yellow cryst. from AcOH. M.p. 213°. Spar. sol. boiling EtOH. Insol. Et_2O.

Duin, Lennep, *Rec. trav. chim.*, 1919, **38**, 368.
Austen, *Ber.*, 1874, **7**, 1248.

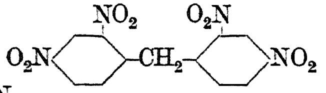
2 : 4 : 6 : 4′-Tetranitrodiphenylamine.
Yellow plates or prisms from toluene. M.p. 223° (219°). Sol. 330 parts toluene at 20°, 200 parts AcOH at 20°. Sol. hot dil. $Na_2CO_3 \longrightarrow$ red sol. Insol. cold EtOH.

Duin, Lennep, *Rec. trav. chim.*, 1919, **38**, 359.

2 : 4 : 2′ : 4′-Tetranitrodiphenylamine.
Reddish-brown plates from EtOH, yellow needles and prisms from AcOH. M.p. 199°. Sol. 44 parts Me_2CO at 17°. Sol. NaOH $\longrightarrow$ scarlet sol. Spar. sol. cold EtOH, AcOH, toluene.

Ryan, Ryan, *Chem. Abstracts*, 1919, **13**, 957.
Carter, *Chem. Zentr.*, 1913, II, 859.
Juillard, *Bull. soc. chim.*, 1905, **33**, 1186.

2 : 4 : 2′ : 4′-Tetranitrodiphenylmethane



$C_{13}H_8O_8N_4$ MW, 348

Yellow prisms from AcOH. M.p. 173°.

Matsumura, *J. Am. Chem. Soc.*, 1929, **51**, 817.

1 : 1 : 2 : 2-Tetranitroethane (sym.-*Tetranitroethane*)

$(O_2N)_2HC{\cdot}CH(NO_2)_2$

$C_2H_2O_8N_4$ MW, 210

Di-K salt: $C_2O_8N_4K_2$. MW, 286. Yellow prisms with metallic reflex from MeOH.Aq. Explodes at 268° (275°) or by percussion. Spar. sol. cold H_2O. Insol. MeOH, EtOH, AcOH. Aq. sol. decomp. on boiling. Cold dil. $H_2SO_4 \longrightarrow$ dinitromethane. $HNO_3 + H_2SO_4 \longrightarrow$ hexanitroethane.

Hunter, *J. Chem. Soc.*, 1924, **125**, 1483.
Will, *Ber.*, 1914, **47**, 963.

Tetranitromethane

$C(NO_2)_4$

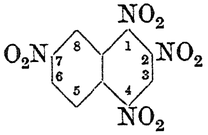
CO_8N_4 MW, 196

F.p. 13° (12·5°). B.p. 125·7°, 21–3°/22 mm. (34–5°/20 mm.). Misc. with EtOH, Et_2O. Insol. H_2O. $D_4^{21\cdot2}$ 1·6377. $n_\alpha^{21\cdot2}$ 1·43416. Heat of comb. C_v 89·6 Cal. Gives yellow col. with cyclopropane derivs. and unsaturated comps.

McKie, *J. Soc. Chem. Ind.*, 1925, **44**, 430T.
Berger, *Compt. rend.*, 1910, **151**, 814.
Hammick, Young, *J. Chem. Soc.*, 1936, 1464.

N : 2 : 4 : 6-Tetranitro-N-methylaniline.
See Tetryl.

1 : 2 : 4 : 7-Tetranitronaphthalene



$C_{10}H_4O_8N_4$ MW, 308

Pale yellow cryst. M.p. 215° decomp.

Chatt, Wynne, *J. Chem. Soc.*, 1943, 33.

1 : 2 : 5 : 8-Tetranitronaphthalene.
Needles from ethyl benzoate, prisms from conc. HNO_3. Decomp. at 270–310°. Spar. sol. EtOH, Me_2CO, AcOH, $CHCl_3$. Ox. $\longrightarrow$ 3 : 6-dinitrophthalic acid. Red. $\longrightarrow$ naphthazarin.

Will, *Ber.*, 1895, **28**, 369.

1 : 2 : 6 : 8-Tetranitronaphthalene.
Pale brown needles from Ac_2O. M.p. 138°.

Chatt, Wynne, *J. Chem. Soc.*, 1943, 33.

1 : 3 : 5 : 7-Tetranitronaphthalene.
Orange-yellow cryst. M.p. 260°.

Chatt, Wynne, *J. Chem. Soc.*, 1943, 33.

1 : 3 : 5 : 8-Tetranitronaphthalene.
Pale yellow tetrahedra from Me_2CO. M.p. 194–5°. Very sol. Me_2CO, conc. HNO_3. Spar. sol. EtOH, AcOH, $CHCl_3$. Sol. alkalis $\longrightarrow$ red sols. Ox. $\longrightarrow$ 3 : 6-dinitrophthalic acid.

1-*Naphthylamine add. comp.*: black needles from C_6H_6. Decomp. at 162°.

2-*Naphthylamine add. comp.*: bronze-green needles. M.p. 163–4°.

Will, *Ber.*, 1895, **28**, 369.
Dhar, *J. Chem. Soc.*, 1920, **117**, 1004.

1 : 3 : 6 : 8-Tetranitronaphthalene.
Yellow needles from EtOH or C_6H_6. M.p. 207°. Explodes on strong heating. Dil. $HNO_3 \longrightarrow$ 3 : 5-dinitrophthalic acid.

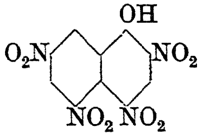
Naphthalene add. comp.: yellow needles from C_6H_6. M.p. 191–2°.

1-*Naphthylamine add. comp.*: purple needles from C_6H_6. M.p. 204–5°.

2-*Naphthylamine add. comp.*: brown plates from C_6H_6. M.p. 211–12°.

Will, *Ber.*, 1895, **28**, 369.
Dhar, *J. Chem. Soc.*, 1920, **117**, 1004.

2 : 4 : 5 : 7-Tetranitro-1-naphthol

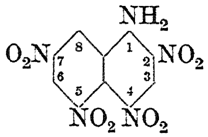


$C_{10}H_4O_9N_4$ MW, 324

Yellow plates or needles from AcOH. M.p. 180°. Sol. 220 parts C_6H_6 at 18°. Spar. sol. cold AcOH. Gives cryst. Na, K, Ag, Ca and Ba salts.

Merz, Weith, *Ber.*, 1882, **15**, 2714.

2 : 4 : 5 : 7-Tetranitro-1-naphthylamine



$C_{10}H_5O_8N_5$ MW, 323

Pale yellow needles from EtOH or C_6H_6. M.p. 194°. Spar. sol. warm EtOH, C_6H_6.

N-Phenyl: $C_{16}H_9O_8N_5$. MW, 399. Orange-yellow needles + C_6H_6 from C_6H_6, dark red needles from EtOH. M.p. 162·5°. Sol. C_6H_6. Spar. sol. warm EtOH, Et_2O. Boiling NaOH.Aq. ⟶ 2 : 4 : 5 : 7-tetranitro-1-naphthol.

Merz, Weith, *Ber.*, 1882, **15**, 2717.

2 : 4 : 5 : 8-Tetranitro-1-naphthylamine.

Yellow needles from EtOH. M.p. 202°.

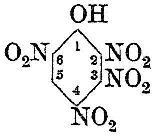
N-Phenyl: orange needles + C_6H_6 from C_6H_6, dark red needles from EtOH. M.p. 253°. Spar. sol. warm EtOH, C_6H_6.

Merz, Weith, *Ber.*, 1882, **15**, 2717.

Tetranitrophenetole.

See under Tetranitrophenol.

2 : 3 : 4 : 6-Tetranitrophenol



$C_6H_2O_9N_4$ MW, 274

Pale yellow cryst. from $CHCl_3$. M.p. 140°.

Me ether: 2 : 3 : 4 : 6 - tetranitroanisole. $C_7H_4O_9N_4$. MW, 288. Leaflets. M.p. 94°. Spar. sol. H_2O, EtOH, Et_2O.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 256.
Claessen, D.R.P., 289,446, (*Chem. Zentr.*, 1916, I, 240).

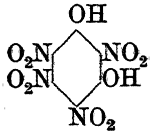
2 : 3 : 5 : 6-Tetranitrophenol.

Me ether: 2 : 3 : 5 : 6-tetranitroanisole. Exists in two forms. (i) Cryst. from EtOH. M.p. 154°. (ii) Pale yellow needles from C_6H_6. M.p. 112°.

Et ether: 2 : 3 : 5 : 6-tetranitrophenetole. $C_8H_6O_9N_4$. MW, 302. Yellow cryst. from EtOH. M.p. 115°.

Blanksma, *Rec. trav. chim.*, 1904, **23**, 114; 1905, **24**, 42.
Devergnes, *Chem. Abstracts*, 1929, **23**, 4207.

Tetranitroresorcinol



$C_6H_2O_{10}N_4$ MW, 290

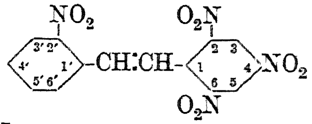
Cryst. from $CHCl_3$ or CCl_4. M.p. 152°. Bitter taste. Explosive.

Mono-Me ether: $C_7H_4O_{10}N_4$. MW, 304. Cryst. from $CHCl_3$ or CCl_4. M.p. 115°. Bitter taste. Explosive.

Mono-Et ether: $C_8H_6O_{10}N_4$. MW, 318. Cryst. from $CHCl_3$ or CCl_4. M.p. 110°. Bitter taste. Explosive.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 35.

2 : 4 : 6 : 2′-Tetranitrostilbene



$C_{14}H_8O_8N_4$ MW, 360

Pale brown needles from EtOH. M.p. 181°.

Bishop, Brady, *J. Chem. Soc.*, 1922, 2367.

2 : 4 : 6 : 3′-Tetranitrostilbene.

Lemon-yellow leaflets from Me_2CO–C_6H_6. M.p. 159°.

Bishop, Brady, *J. Chem. Soc.*, 1922, 2367.

2 : 4 : 6 : 4′-Tetranitrostilbene.

Yellow needles. M.p. 196°. Sol. AcOH. Spar. sol. EtOH, C_6H_6.

Ullmann, Gschwind, *Ber.*, 1908, **41**, 2297.

2 : 4 : 2′ : 4′-Tetranitrostilbene.

Yellow needles from AcOH or $PhNO_2$. M.p. 266–7° decomp. Insol. Et_2O.

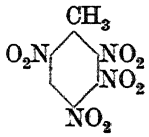
Escales, *Ber.*, 1904, **37**, 3599.
Green, Baddiley, *J. Chem. Soc.*, 1908, **93**, 1725.

2 : 6 : 2′ : 6′-Tetranitrostilbene.

Yellow needles from $PhNO_2$. M.p. 250°. Sol. Me_2CO. Insol. EtOH, Et_2O, ligroin.

Reich, Wetter, Widmer, *Ber.*, 1912, **45**, 3059.

2 : 3 : 4 : 6-Tetranitrotoluene



$C_7H_4O_8N_4$ MW, 272

Cryst. from $CHCl_3$ or conc. HNO_3. M.p. 136·5°.

Holleman, *Rec. trav. chim.*, 1930, **49**, 501.

Tetraphenylbenzanthracene.

See Rubrene.

Tetraphenyldiarsine.

See Phenylcacodyl.

***sym.*-Tetraphenylethane**

$(C_6H_5)_2CH\cdot CH(C_6H_5)_2$

$C_{26}H_{22}$ MW, 334

Prisms from $CHCl_3$. M.p. 211°. B.p. 358–62°, 260°/16 mm.

Montagne, *Rec. trav. chim.*, 1906, **25**, 407.
Morris, Thomas, Brown, *Ber.*, 1910, **43**, 2959.
Nauta, Mulder, *Rec. trav. chim.*, 1939, **58**, 1062.

***unsym.*-Tetraphenylethane** (*α-Benzyltritan, triphenylbenzylmethane*)

$C_6H_5\cdot CH_2\cdot C(C_6H_5)_3$

$C_{26}H_{22}$ MW, 334

Plates from AcOEt. M.p. 144°. B.p. 277–80°/21 mm. Mod. sol. C_6H_6. Spar. sol. EtOH, Et_2O.

Gomberg, Cone, *Ber.*, 1906, **39**, 1463.

1 : 1 : 2 : 2 - Tetraphenylethyl Alcohol (*Hydroxy*-sym.-*tetraphenylethane*, *α-benzhydrylbenzhydrol*)

$(C_6H_5)_2CH\cdot C(C_6H_5)(OH)(C_6H_5)$

$C_{26}H_{22}O$ MW, 350

Needles from C_6H_6. M.p. 326° (232·5–233°, 244°). Mod. sol. EtOH, Et_2O, C_6H_6, ligroin.
Benzoyl: prisms from AcOEt. M.p. 155°.
Phenylurethane: cryst. from C_6H_6–pet. ether. M.p. 163–5°.

Richard, *Compt. rend.*, 1934, **198**, 1242.
Bergmann, Wagenberg, *Ber.*, 1930, **63**, 2591.
Boyle, McKenzie, Mitchell, *Ber.*, 1937, **70**, 2153.
Banchetti, *Gazz. chim. ital.*, 1942, **72**, 74.

1 : 2 : 2 : 2 - Tetraphenylethyl Alcohol (*ω-Triphenylmethylbenzyl alcohol*, *hydroxy*-unsym.-*tetraphenylethane*)

$(C_6H_5)_3C\cdot CH(C_6H_5)(OH)$

$C_{26}H_{22}O$ MW, 350

Plates from EtOH. M.p. 151°.
Acetyl: cryst. from EtOH. M.p. 131°.

Schlenk, Ochs, *Ber.*, 1916, **49**, 611.

Tetraphenylethylene

$(C_6H_5)_2C{:}C(C_6H_5)_2$

$C_{26}H_{20}$ MW, 332

Plates from EtOH–C_6H_6. M.p. 223–4° (220–1°). B.p. 415–25°. Sol. C_6H_6. Spar. sol. EtOH, Et_2O.

Mackenzie, *J. Chem. Soc.*, 1922, **121**, 1697.

Tetraphenylethylene Glycol.
See Benzpinacol.
Tetraphenylethylene oxide.
See Benzpinacolin.
Tetraphenylfuran.
See Lepidene.

1 : 1 : 3 : 3-Tetraphenylguanidine

$(C_6H_5)_2N^3\cdot C^2(:N^4H)\cdot N^1(C_6H_5)_2$

$C_{25}H_{21}N_3$ MW, 363

Cryst. from ligroin. M.p. 130–1°. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.
4-N-*Benzoyl*: cryst. from EtOH. M.p. 142–4°.

Weith, *Ber.*, 1874, **7**, 843.
Johnson, Chernoff, *J. Am. Chem. Soc.*, 1912, **34**, 170.

1 : 1 : 3 : 4-Tetraphenylguanidine

$C_6H_5\cdot NH\cdot C(:N\cdot C_6H_5)\cdot N(C_6H_5)_2$

$C_{25}H_{21}N_3$ MW, 363

Cryst. from EtOH. M.p. 137–40°.
Chloroplatinate: m.p. 240–2°.

Steindorff, *Ber.*, 1904, **37**, 964.

Tetraphenylhydrazine

$(C_6H_5)_2N\cdot N(C_6H_5)_2$

$C_{24}H_{20}N_2$ MW, 336

Prisms from $CHCl_3$–EtOH. M.p. 147° decomp. Sol. Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. EtOH.

Chattaway, Ingle, *J. Chem. Soc.*, 1895, **67**, 1091.
Wieland, Gambarjan, *Ber.*, 1906, **39**, 1500.

1 : 1 : 2 : 3-Tetraphenylisopropyl Alcohol (2-*Hydroxy*-1 : 1 : 2 : 3-*tetraphenylpropane*)

$C_6H_5\cdot CH_2\cdot C(C_6H_5)(OH)\cdot CH(C_6H_5)_2$

$C_{27}H_{24}O$ MW, 364

Needles from ligroin. M.p. 135–6°. Sol. EtOH, C_6H_6. Mod. sol. ligroin.
Acetyl: needles from C_6H_6. M.p. 151–3°.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1489.
Orékhoff, *Bull. soc. chim.*, 1919, **25**, 186.

Tetraphenylmethane

$C(C_6H_5)_4$

$C_{25}H_{20}$ MW, 320

Cryst. from C_6H_6. M.p. 282°. B.p. 431°. Insol. Et_2O, AcOH, ligroin. Sublimes in needles. Sol. H_2SO_4 with red col.

Gomberg, Kamm, *J. Am. Chem. Soc.*, 1917, **39**, 2009.
Gomberg, Cone, *Ber.*, 1906, **39**, 1463.
Seibert, Bergstrom, *J. Org. Chem.*, 1945, **10**, 544.

Tetraphenylphenylenediamine.
See under Phenylenediamine.
Tetraphenylphosphonium halides.
See under Triphenylphosphine.

1 : 1 : 1 : 3-Tetraphenylpropane

$C_6H_5{\cdot}CH_2{\cdot}CH_2{\cdot}C(C_6H_5)_3$

$C_{27}H_{24}$ MW, 348

Prisms from EtOH. M.p. 126°.

Wieland, Kloss, *Ann.*, 1929, **470**, 214.

1 : 1 : 2 : 3-Tetraphenylpropane

$C_6H_5{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}CH(C_6H_5)_2$

$C_{27}H_{24}$ MW, 348

Needles from EtOH. M.p. 87–9°.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1491.

1 : 1 : 3 : 3-Tetraphenylpropane

$(C_6H_5)_2CH{\cdot}CH_2{\cdot}CH(C_6H_5)_2$

$C_{27}H_{24}$ MW, 348

Needles from EtOH. M.p. 139°. Sol. Et_2O, $CHCl_3$, C_6H_6.

Wittig, Obermann, *Ber.*, 1934, **67**, 2056.
Vorländer, Siebert, *Ber.*, 1906, **39**, 1028.

1 : 1 : 2 : 3-Tetraphenylpropyl Alcohol (*1-Hydroxy*-1 : 1 : 2 : 3-*tetraphenylpropane*)

$C_6H_5{\cdot}CH_2{\cdot}CH(C_6H_5){\cdot}C(OH)(C_6H_5)_2$

$C_{27}H_{24}O$ MW, 364

Needles from EtOH. M.p. 165°.

Sernagiotto, *Chem. Abstracts*, 1920, **14**, 1672.
McKenzie, Winton, *J. Chem. Soc.*, 1940, 840.

1 : 1 : 3 : 3-Tetraphenylpropyl Alcohol (*1-Hydroxy*-1 : 1 : 3 : 3-*tetraphenylpropane*)

$(C_6H_5)_2CH{\cdot}CH_2{\cdot}C(OH)(C_6H_5)_2$

$C_{27}H_{24}O$ MW, 364

Needles. M.p. 95–6°. Sol. EtOH, Et_2O. Spar. sol. ligroin.

Kohler, *Am. Chem. J.*, 1904, **31**, 651.

1 : 2 : 2 : 3-Tetraphenylpropyl Alcohol (*1-Hydroxy*-1 : 2 : 2 : 3-*tetraphenylpropane*)

$C_6H_5{\cdot}CH_2{\cdot}C(C_6H_5)_2{\cdot}CH(OH){\cdot}C_6H_5$

$C_{27}H_{24}O$ MW, 364

Cryst. from pet. ether. M.p. 141–2°.

Schlenk *et al.*, *Ann.*, 1928, **463**, 261.

1 : 2 : 3 : 3-Tetraphenylpropyl Alcohol (*1-Hydroxy*-1 : 2 : 3 : 3-*tetraphenylpropane*)

$(C_6H_5)_2CH{\cdot}CH(C_6H_5){\cdot}CH(OH){\cdot}C_6H_5$

$C_{27}H_{24}O$ MW, 364

Cryst. from Et_2O. M.p. 132°. Sol. EtOH, AcOH. Mod. sol. Et_2O.

Japp, Klingemann, *J. Chem. Soc.*, 1890, **57**, 669.

1 : 1 : 2 : 3-Tetraphenylpropylene (1 : 1 : 2-*Triphenyl-2-benzylethylene*)

$C_6H_5{\cdot}CH_2{\cdot}C(C_6H_5){:}C(C_6H_5)_2$

$C_{27}H_{22}$ MW, 346

Needles from methyl ethyl ketone. M.p. 142°. Sol. $CHCl_3$, C_6H_6, CCl_4. Spar. sol. Et_2O. Prac. insol. cold EtOH, AcOH.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1489.
Meisenheimer, Schlichenmaier, *Ann.*, 1927, **456**, 151.

1 : 1 : 3 : 3-Tetraphenylpropylene

$(C_6H_5)_2CH{\cdot}CH{:}C(C_6H_5)_2$

$C_{27}H_{22}$ MW, 346

Cryst. from EtOH. M.p. 127–8°. Sol. Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. pet. ether. Sol. conc. H_2SO_4 with yellow col.

Wittig, Obermann, *Ber.*, 1934, **67**, 2054.
Vorländer, Siebert, *Ber.*, 1906, **39**, 1032.

1 : 2 : 3 : 3-Tetraphenylpropylene

$(C_6H_5)_2CH{\cdot}C(C_6H_5){:}CH{\cdot}C_6H_5$

$C_{27}H_{22}$ MW, 346

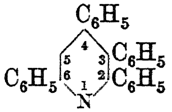
Needles from EtOH. M.p. 131°.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1489.

Tetraphenylpyrazine.

See Amaron.

2 : 3 : 4 : 6-Tetraphenylpyridine



$C_{29}H_{21}N$ MW, 383

Needles from EtOH. M.p. 182°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Insol. ligroin.

Picrate: yellow needles from Et_2O. M.p. 192°.

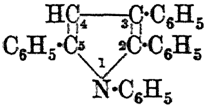
Dilthey, Böttler, *Ber.*, 1919, **52**, 2048.
Dilthey, Nüsslein, Meyer, Kaffer, *J. prakt. Chem.*, 1922, **104**, 33.

2 : 3 : 5 : 6-Tetraphenylpyridine.

Prisms from EtOH–C_6H_6. M.p. 233·5°. Sol. $CHCl_3$, CS_2. Mod. sol. EtOH, Me_2CO, C_6H_6. Spar. sol. Et_2O, AcOH, ligroin. Sol. conc. H_2SO_4 with blue fluor.

Carpenter, *Ann.*, 1898, **302**, 233.

1 : 2 : 3 : 5-Tetraphenylpyrrole



$C_{28}H_{21}N$ MW, 371

Needles from AcOH. M.p. 197°. Sol. Et_2O, C_6H_6. Mod. sol. EtOH, AcOH.

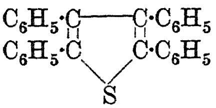
Smith, *J. Chem. Soc.*, 1890, **57**, 646.

2 : 3 : 4 : 5-Tetraphenylpyrrole.
Needles from AcOH. M.p. 214–15°. Prac. insol. EtOH. Sol. conc. H_2SO_4 with yellow col.
N-*Me*: $C_{29}H_{23}N$. MW, 385. Plates from Et_2O. M.p. 214°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.
N-*Et*: $C_{30}H_{25}N$. MW, 399. Needles from $CHCl_3$. M.p. 221°.
N-*Acetyl*: needles from AcOH or C_6H_6. M.p. 226°. Prac. insol. EtOH.

Robinson, Robinson, *J. Chem. Soc.*, 1918, **113**, 644.
Fehrlin, *Ber.*, 1889, **22**, 555.
Davidson, *J. Org. Chem.*, 1939, **3**, 361.

Tetraphenylsilane.
See Silicon tetraphenyl.

Tetraphenylthiophene (*Thionessal*)



$C_{28}H_{20}S$ MW, 388

Needles from EtOH. M.p. 184–5°. B.p. about 400°. Mod. sol. Et_2O, CS_2, C_6H_6. Spar. sol. ligroin. Very spar. sol. EtOH.

Bergman, *J. Chem. Soc.*, 1936, 505.
Dilthey *et al.*, *Ber.*, 1935, **68**, 1159.
Smith, Hoehn, *J. Am. Chem. Soc.*, 1941, **63**, 1184.

Tetraphenylthiourea.
See under Thiourea.

Tetraphenylurea.
See under Urea.

Tetraphthene.
See Tetrahydroacenaphthene.

Tetratriacontane

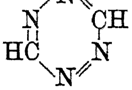
$CH_3\cdot[CH_2]_{32}\cdot CH_3$

$C_{34}H_{70}$ MW, 478

Obtained by electrolysis of potassium stearate. Plates from Et_2O. M.p. 72·6°. B.p. 285·4°/3 mm. D_4^{90} 0·7728. n_D^{90} 1·4296.

Garner, Bibber, King, *J. Chem. Soc.*, 1931, 1533.
Mazee, *Rec. trav. chim.*, 1948, **67**, 197.

1 : 2 : 4 : 5-Tetrazine

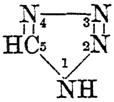


$C_2H_2N_4$ MW, 82

Crimson cryst. M.p. 99°. Sol. H_2O, EtOH, Et_2O, liq. NH_3. Sol. H_2SO_4 with red col. Sublimes. Volatile at room temp.

Wood, Bergstrom, *J. Am. Chem. Soc.*, 1933, **55**, 3649.
Curtius, Darapsky, Müller, *Ber.*, 1907, **40**, 84.

1 : 2 : 3 : 4-Tetrazole (*Pyrro-αββ'-triazole*)



CH_2N_4 MW, 70

Plates from EtOH. M.p. 156°. Sol. H_2O, EtOH, Me_2CO, AcOH. Spar. sol. Et_2O, C_6H_6. Sublimes. Salts are explosive.
N-*Me*: *see* 1-Methyl-1 : 2 : 3 : 4-tetrazole.
N-*Phenyl*: *see* 1-Phenyl-1 : 2 : 3 : 4-tetrazole.

Freund, Paradies, *Ber.*, 1901, **34**, 3110.
Pechmann, Wedekind, *Ber.*, 1895, **28**, 1693.

Tetrolaldehyde (*Allylene-1-aldehyde, methylacetylene-aldehyde*)

$CH_3\cdot C⋮C\cdot CHO$

C_4H_4O MW, 68

F.p. − 26°. B.p. 106·5–107°, 27–8°/34 mm. D_0^0 0·944, D_0^{17} 0·9265. n_D^{17} 1·4467. Readily oxidises in air.
Oxime: needles. M.p. 108–9°.
Semicarbazone: m.p. 158°.
Hydrazone: liq. B.p. 170° decomp., 63–5°/15 mm. $D_0^{18\cdot5}$ 0·9768. $n_D^{18\cdot5}$ 1·530. Misc. with H_2O, EtOH, Et_2O.
p-*Nitrophenylhydrazone*: yellow needles from EtOH.Aq. M.p. 157–8°.
Azine: yellow needles from EtOH. M.p. 123–4°.
Di-Me acetal: b.p. 144·5°. D^{15} 0·954.
Di-Et acetal: b.p. 169–70°, 62–5°/15 mm. D_0^{16} 0·9012. n_D^{16} 1·4269.

Claisen, *Ber.*, 1911, **44**, 1166.
Viguier, *Compt. rend.*, 1911, **152**, 1491.

Tetrolic Acid (*Methylacetylene-carboxylic acid, methylpropiolic acid, allylene-1-carboxylic acid*)

$CH_3\cdot C⋮C\cdot COOH$

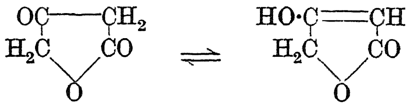
$C_4H_4O_2$ MW, 84

Plates from Et_2O or CS_2. M.p. 77–8°. B.p. 203°, 99–100°/18 mm. $k = 2{\cdot}46 \times 10^{-3}$ at 25°. Sol. H_2O, EtOH, Et_2O, $CHCl_3$, CS_2. Volatile in steam. At 211° ⟶ allylene.
Et ester: $C_6H_8O_2$. MW, 112. Oil. B.p. 163–4°. $D_4^{24\cdot1}$ 0·9621. $n_{He}^{24\cdot1}$ 1·43495.
Chloride: C_4H_3OCl. MW, 102·5. Liq. Fumes in air. Very unstable.
Amide: C_4H_5ON. MW, 83. Cryst. from EtOH. M.p. 147–8°. Mod. sol. H_2O, EtOH. Spar. sol. Et_2O. Volatile in steam.

Feist, *Ann.*, 1906, **345**, 104.
Auwers, *Ber.*, 1935, **68**, 1637.
Bourguel, Yvon, *Bull. soc. chim.*, 1929, **45**, 1067.

Tetronal.
See Ethylsulphonal.

Tetronic Acid (*Dihydroxycrotonic lactone, 3-hydroxyacetoacetic lactone, 2-ketobutyrolactone, 2 : 4-diketotetrahydrofuran*)



$C_4H_4O_3$ MW, 100

Plates from EtOH–ligroin. Sinters at 135°. M.p. 141°. Very sol. H_2O, warm EtOH. Spar. sol. Et_2O, $CHCl_3$, ligroin, C_6H_6. Strong monobasic acid. Decomposes bicarbonates. $FeCl_3$ $\longrightarrow$ dark red col. Heat conc. aq. sol. $\longrightarrow$ anhydrotetronic acid.

Anhydride: anhydrotetronic acid. $C_8H_6O_5$. MW, 182. Needles + $1H_2O$ from hot H_2O. M.p. 263° decomp. Sol. EtOH. Spar. sol. cold H_2O, Et_2O, $CHCl_3$, C_6H_6. Decomposes bicarbonates. $FeCl_3$ $\longrightarrow$ red. col.

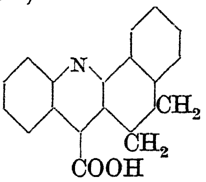
Oxime: plates from hot EtOH. Decomp. at 146°. Very sol. H_2O. Sol. EtOH, Et_2O. No col. with $FeCl_3$.

Benzoyl: plates or prisms from $CHCl_3$–ligroin. M.p. 120°.

Phenylhydrazone: prisms from EtOH.Aq. M.p. 128°.

Wolff, Schwabe, *Ann.*, 1896, **291**, 234.

Tetrophane (*3 : 4-Dihydro-1 : 2-benzacridine-14-carboxylic acid*)



$C_{18}H_{13}O_2N$ MW, 275

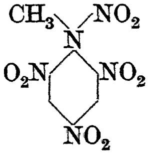
Yellow needles. M.p. 252° decomp. to 3 : 4-dihydro-1 : 2-naphthacridine. Very sol. tetralin. Mod. sol. AcOH. Spar. sol. EtOH. Insol. Et_2O, C_6H_6.

Et ester: $C_{20}H_{17}O_2N$. MW, 303. Needles. M.p. 80°.

Amide: $C_{18}H_{14}ON_2$. MW, 274. Colourless powder from EtOH–pet. ether. M.p. 220–2°.

v. Braun, Wolff, *Ber.*, 1922, **55**, 3679.

Tetryl (*N-Nitro-N-methyl-2 : 4 : 6-trinitroaniline, methylpicrylnitramine*)

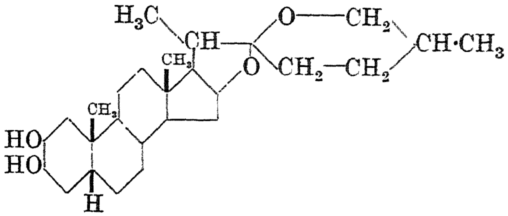


$C_7H_5O_8N_5$ MW, 287

Yellow prisms from EtOH. M.p. 131–2°. D^{19} 1·57. Sol. Me_2CO, Py. Mod. sol. AcOEt, C_6H_6. Spar. sol. MeOH, EtOH. Explodes on heating to about 180–90°. NaOH $\longrightarrow$ picric acid. Used as explosive chiefly in admixture with other explosives such as trinitrotoluene (T.N.T.).

van Duin, *Rec. trav. chim.*, 1918, **37**, 112.
Tanner, *Chem. Met. Eng.*, 1923, **29**, 404.
Hodgson, Turner, *J. Chem. Soc.*, 1942, 584.
Boyer, Spencer, Wright, *Can. J. Research*, 1946, **24***B*, 200.

Texogenin



Suggested structure

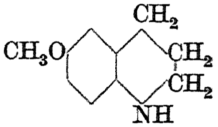
$C_{27}H_{44}O_4$ MW, 432

Isolated from *Yucca schottii*, Engelm. Cryst. from Me_2CO. M.p. 172–5°.

Acetyl deriv.: needles from MeOH. M.p. 172–3°.

Marker *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2167.

Thalline (*6-Methoxy-1 : 2 : 3 : 4-tetrahydroquinoline*)



$C_{10}H_{13}ON$ MW, 163

Rhombic cryst. from H_2O, prisms from EtOH or pet. ether, m.p. 42–3°. B.p. 283°/735 mm., 127–30°/1 mm. n^{50} 1·5718. Very sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O, pet. ether. Difficultly volatile in steam. HCl, H_2SO_4 and AcOH salts have antipyretic action. Ox. agents $\longrightarrow$ emerald green col. $FeCl_3$ on boiling $\longrightarrow$ brownish-green or rose col.

B_2,HI: prisms from EtOH. M.p. 155–6°.

N-*Me*: $C_{11}H_{15}ON$. MW, 177. Oil. B.p. 227–228·5°, 150–1°/10 mm. Dil. HCl sol. + $FeCl_3$ $\longrightarrow$ dark red col. Cl water $\longrightarrow$ reddish-yellow col. *Methiodide*: needles + $1H_2O$ from H_2O, prisms from EtOH. M.p. 223–4° decomp. *Picrate*: yellow plates. M.p. 164°.

N-*Et*: $C_{12}H_{17}ON$. MW, 191. Yellow oil. B.p. 287–287·5° slight decomp. Misc. with EtOH, Et_2O. Very sol. min. acids. Insol. H_2O. *Ethiodide*: needles. M.p. 131–3°.

N-*Allyl*: $C_{13}H_{17}ON$. MW, 203. Yellow oil. B.p. 176°/12 mm.

N-*Acetyl*: prisms from Et_2O–pet. ether. M.p. 46–7°.

Picrate: yellow needles. M.p. 162° (164°).

Skraup, *Monatsh.*, 1885, **6**, 767.
Cromwell, Caughlan, Gilbert, *J. Am. Chem. Soc.*, 1944, **66**, 401.

Thallium triethyl

$Tl(C_2H_5)_3$

$C_6H_{15}Tl$ MW, 291

Deep yellow heavy oil. B.p. 50–1°/1·5 mm. Decomp. in light. Burns with green flame.

Birch, *J. Chem. Soc.*, 1934, 1132.
Rochow, Dennis, *J. Am. Chem. Soc.*, 1935, **67**, 486.

Thallium tri-isobutyl

$Tl(CH_2{\cdot}CH(CH_3)_2)_3$

$C_{12}H_{27}Tl$ MW, 375

Pale lemon yellow liq. B.p. 74–6°/1·6 mm. slight decomp. Decomp. in light.

Birch, *J. Chem. Soc.*, 1934, 1132.

Thallium trimethyl

$Tl(CH_3)_3$

C_3H_9Tl MW, 249

Long colourless needles. M.p. 38·5°. B.p. 85°/76 mm., 66·5°/60 mm., 57°/40 mm. Explodes above 90°. Spontaneously inflammable in air. Misc. with Et_2O, C_6H_6.

Gilman, Jones, *J. Am. Chem. Soc.*, 1946, **68**, 517.

Thallium triphenyl

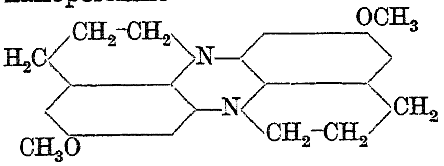
$Tl(C_6H_5)_3$

$C_{18}H_{15}Tl$ MW, 435

Needles. M.p. 188–9°. Decomp. at 215–16°. Sol. C_6H_6. Insol. pet. ether.

Birch, *J. Chem. Soc.*, 1934, 1132.

Thalloperazine

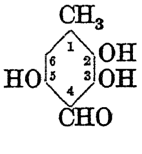


$C_{20}H_{22}O_2N_2$ MW, 322

Yellow cryst. from AcOEt. M.p. 160°. Sol. C_6H_6. Mod. sol. Et_2O. Spar. sol. AcOEt, cold EtOH. H_2SO_4 ⟶ red sol. with reddish-green fluorescence; with H_2O, violet ⟶ pink.

Wieland, Haas, *Ber.*, 1920, **53**, 1342.

Thamnol (2 : 3 : 5-*Trihydroxy*-p-*toluic aldehyde*)



$C_8H_8O_4$ MW, 168

Yellow prisms from C_6H_6. M.p. 186°. Sol. EtOH, Et_2O, AcOEt, Py. Spar. sol. cold C_6H_6. Sol. H_2O ⟶ acid sol. Sol. alkalis ⟶ intense yellow col. Alc. $FeCl_3$ ⟶ green col.

3 : 5-*Di-Me ether*: $C_{10}H_{12}O_4$. MW, 196. Yellow prisms from C_6H_6. M.p. 104°.

Triacetyl: needles from EtOH. M.p. 133°.

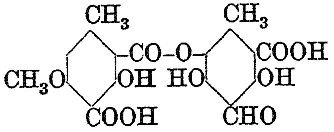
Anil: brownish leaflets. M.p. 128–9°.

Phenylhydrazone: brownish-red spears from EtOH. M.p. 194° decomp.

p-*Nitrophenylhydrazone*: brownish-violet needles from EtOH.Aq. M.p. about 320° decomp.

Koller, Hamburg, *Monatsh.*, 1935, **65**, 378.
Asahina, Fuzikawa, *Ber.*, 1932, **65**, 58.
Asahina, Ihara, *Ber.*, 1929, **62**, 1204.

Thamnolic Acid



$C_{19}H_{16}O_{11}$ MW, 420

Constituent of many lichens. Colourless prisms from Me_2CO. M.p. 223°. Sol. alkalis and alk. carbonates ⟶ yellow sols. Alc. $FeCl_3$ ⟶ brownish-red col. Loses CO_2 on heating to 120–30° with Me_2CO.

Di-Me ester: yellow prisms from C_6H_6–EtOH. M.p. 158°.

Anil: orange-yellow cryst. from EtOH. M.p. 206–8°.

Phenylhydrazone: yellow prisms from EtOH. M.p. 173–4°.

p-*Nitrophenylhydrazone*: orange-yellow cryst. from EtOH. M.p. 238–9°.

Asahina, Hiraiwa, *Ber.*, 1936, **69**, 330.
Asahina, Ihara, *Ber.*, 1929, **62**, 1196.
Asahina, Aoki, Fuzikawa, *Ber.*, 1941, **74**, 824.

Thapsic Acid (*Thapsiaic acid*, 1 : 14-*tetradecane-dicarboxylic acid*)

$HOOC{\cdot}[CH_2]_{14}{\cdot}COOH$

$C_{16}H_{30}O_4$ MW, 286

Occurs as ester in roots of *Thapsia garganica*, Linn., and in wax of *Juniperus sabina*. Plates from EtOH or AcOEt. M.p. 126°. Sol. EtOH. Spar. sol. Et_2O. Insol. H_2O, CS_2, C_6H_6. Distils undecomp. Ac_2O ⟶ anhydride.

Mono-Me ester: $C_{17}H_{32}O_4$. MW, 300. Cryst. from MeOH. M.p. 45–6°.

Di-Me ester: $C_{18}H_{34}O_4$. MW, 314. M.p. 51–2°. B.p. 150–60°/0·3 mm.

Mono-Et ester: $C_{18}H_{34}O_4$. MW, 314. B.p. 220°/3 mm.

Di-Et ester: $C_{20}H_{38}O_4$. MW, 342. Cryst. M.p. 39°. B.p. 160–5°/0·3 mm. Very sol. Et_2O, EtOH. Spar. sol. pet. ether.

Di-n-*butyl ester*: m.p. 33°.

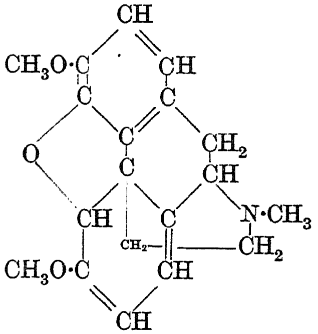
Anhydride: $C_{16}H_{28}O_3$. MW, 268. Cryst. powder from C_6H_6. M.p. 71°. Boiling H_2O ⟶ thapsic acid.

Dianilide: cryst. from MeOH. M.p. 163°.

Schmid, Kemeny, *Monatsh.*, 1935, **66**, 3.
Bougault, *Compt. rend.*, 1910, **150**, 875.
Canzoneri, *Gazz. chim. ital.*, 1883, **13**, 516.

Thapsin.
See Calycopterin.

Thebaine (*Methyl ether of enol of codeinone*)



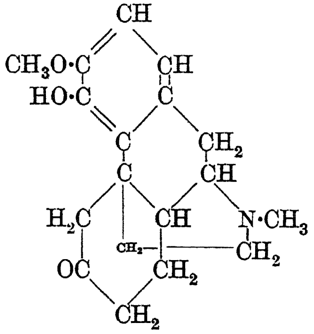
$C_{19}H_{21}O_3N$ MW, 311

Plates from EtOH.Aq., prisms from EtOH. M.p. 193°. Sublimes at 91°/0·01 mm. Very sol. EtOH, $CHCl_3$. Sol. 140 parts Et_2O at 10°, 18 parts cold C_6H_6. Sol. H_2SO_4 ⟶ red col. Tasteless. $[\alpha]_D$ − 218·6° in EtOH. Decomp. by dil. min. acids. Dil. HCl ⟶ thebenin. Very poisonous.

Picrate: m.p. 217°.

v. Braun, Cahn, *Ann.*, 1926, **451**, 55.
Schöpf, Winterholder, *Ann.*, 1927, **452**, 232.

Thebainol



$C_{18}H_{23}O_3N$ MW, 301

Cryst. + MeOH from MeOH, m.p. 50–4°; colourless prisms from Et_2O, m.p. 135–6°. $[\alpha]_D^{25}$ + 67·05° in EtOH. Sol. dil. alkalis. Forms cryst. Na salt. Forms benzylidene and piperonylidene derivs.

Oxime: cryst. from EtOH. M.p. 217–18°. $[\alpha]_D^{18}$ + 104·2° in AcOH.Aq.

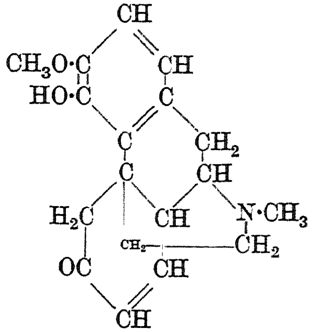
Semicarbazone: needles from EtOH–AcOEt. M.p. 217–18°.

Perchlorate: cryst. from EtOH. M.p. 245°.

Methiodide: prisms from EtOH. M.p. 243° decomp.

Gulland, *J. Chem. Soc.*, 1928, 706.
Schöpf, Borkowsky, *Ann.*, 1927, **458**, 170.
Gulland, Robinson, *J. Chem. Soc.*, 1923, **123**, 998.
Pschorr, *Ber.*, 1905, **38**, 3167.

Thebainone



$C_{18}H_{21}O_3N$ MW, 299

Colourless needles + $\frac{1}{2}H_2O$ from EtOH.Aq. M.p. 151–2°. Loses H_2O at 100° in vacuo. $[\alpha]_D^{220}$ + 9·6° in EtOH. Very sol. Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. Et_2O. Sol. 250 parts cold H_2O, 120 parts hot. Sol. 4 parts EtOH, 5 parts AcOEt. Yellow aq. sol. reacts alkaline. Sol. alkalis ⟶ yellow col. Sol. conc. HCl, H_2SO_4. Forms benzylidene and piperonylidene derivs.

B,HI: needles from H_2O. M.p. 258–9°.

Me ether: $C_{19}H_{23}O_3N$. MW, 313. Prisms from MeOH. M.p. 156°. *Methiodide*: plates from EtOH. M.p. 256°.

Oxime: prisms + $\frac{1}{2}H_2O$ from EtOH.Aq. M.p. 200–1° (185–6°). *B,HCl*: needles from H_2O. M.p. 290–1°.

Semicarbazone: needles from AcOEt. M.p. 227°.

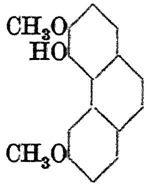
Methiodide: needles from EtOH.Aq. M.p. 255–6° (223°).

Acetyl: prisms from Et_2O–pet. ether. M.p. 100–1°. *Methiodide*: prisms from EtOH. M.p. 223–5°. *Semicarbazone*: needles from EtOH. M.p. 249°. *Phenylhydrazone*: prisms from EtOH. M.p. 225–6°.

Picrate: prisms from EtOH. M.p. 250–3°.

Schöpf, Hirsch, *Ann.*, 1931, **489**, 240.
Gulland, *J. Chem. Soc.*, 1928, 702.
Schöpf, Borkowsky, *Ann.*, 1927, **458**, 148.
Gulland, Robinson, *J. Chem. Soc.*, 1923, 998.
Pschorr, *Ber.*, 1905, **38**, 3163.

Thebaol (*4-Hydroxy-3 : 6-dimethoxyphenanthrene*)



$C_{16}H_{14}O_3$ MW, 254

Plates and columns from AcOH. M.p. 94°. Very sol. EtOH, Et_2O, $CHCl_3$, Me_2CO, C_6H_6. Spar. sol. AcOH, ligroin. Zn dust dist. ⟶ phenanthrene.

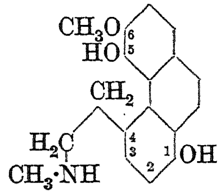
Me ether: methylthebaol. $C_{17}H_{16}O_3$. MW, 268. Thick oil. *Picrate*: reddish-brown needles from EtOH. M.p. 110–12°.

Acetyl: plates from EtOH. M.p. 118–22°. *Picrate*: red needles. M.p. 139°.

Benzoyl: needles from AcOH. M.p. 160–1°.

Pschorr, *Ber.*, 1912, **45**, 2218.
Freund, Göbel, *Ber.*, 1897, **30**, 1371, 1389.

Thebenine (1 : 5-*Dihydroxy*-6-*methoxy*-4-[β-*methylaminoethyl*]-*phenanthrene*)



$C_{18}H_{19}O_3N$ MW, 297

Amorph. Spar. sol. boiling EtOH. Sol. aq. KOH. Insol. Et_2O, C_6H_6. Sol. conc. H_2SO_4 ⟶ blue col. Readily oxidises especially in presence of alkalis. Zn dust dist. ⟶ pyrene.

B,HCl: plates + $3H_2O$. Sinters at 231°. M.p. 235°.

B_2,H_2SO_4: yellow plates + $1H_2O$. Sinters at 205°. M.p. 209–10°.

Oxalate: prisms. M.p. 275–6°.

1-*Me ether*: methebenine. $C_{19}H_{21}O_3N$. MW, 311. Cryst. or amorph. from EtOH. Sinters at 155°. M.p. 165–7°. Pptd. from alk. sol. with CO_2. Hot dil. HCl ⟶ thebenine. *B,HCl*: needles from EtOH. M.p. 250°. *B,HI*: plates from EtOH.Aq. Sinters at 190°. M.p. 195–8°. *Sulphate*: needles. M.p. 238·5°. N : 5-*Diacetyl*: plates from EtOH. M.p. 179°. N : 5-*Dibenzoyl*: needles from EtOH. M.p. 159°.

N-*Me*: *methiodide*: thebeninmethin methiodide. Cryst. from EtOH. M.p. 206–8°.

N : 1-*Di-Me*: $C_{20}H_{23}O_3N$. MW, 325. *Methiodide*: methebeninmethin methiodide. Columns from EtOH.Aq. M.p. 215°.

1 : 5-*Di-Me ether*: *methiodide*: aggregates of needles from EtOH. M.p. 247°. *Methosulphate*: needles from EtOH or Et_2O. Sinters at 268°. M.p. 277°.

1-*Et ether*: ethebenine. $C_{20}H_{23}O_3N$. MW, 325. Yellow, amorph. *B,HCl*: microplates from EtOH.Aq. M.p. 248°. *B,HI*: plates + $1H_2O$ from H_2O. Sinters at 200°. M.p. 206–7°. N : 5-*Diacetyl*: cryst. from EtOH. M.p. 163°.

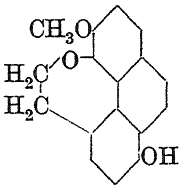
N-*Me*: 1-*Et*: $C_{21}H_{25}O_3N$. MW, 339. *Methiodide*: ethebeninmethin methiodide. Plates from EtOH.Aq. Sinters at 210°. M.p. 215°.

1-*Propyl ether*: prothebenine. $C_{21}H_{25}O_3N$. MW, 339. Needles from EtOH. Sinters at 167°. M.p. 172–3°. *B,HCl*: plates from EtOH. Sinters at 215°. M.p. 221–2°. *B,HI*: plates. M.p. 212–13°.

N : 1 : 5-*Triacetyl*: hydrated needles from EtOH.Aq. M.p. 72–80°. Anhyd. cryst. from abs. EtOH. M.p. 160–1°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 921.
Pschorr, *Ann.*, 1910, **373**, 69.
Freund, Holthof, *Ber.*, 1899, **32**, 179, 181.
Freund, Michaels, *Ber.*, 1897, **30**, 1375.

Thebenol



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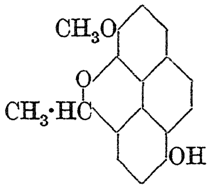


$C_{17}H_{14}O_3$ MW, 266

Rhombohedra from hot AcOH. M.p. 186–8°. Very sol. EtOH, Et_2O. Sol. C_6H_6, KOH.Aq. Insol. H_2O, ligroin, Na_2CO_3, NH_4OH. Alc. sol. reduces $AgNO_3$. Zn dust dist., or heat with HI(+ P) ⟶ pyrene. KOH fusion ⟶ northebenol.

Na salt: cryst. ppt. M.p. about 210–12°.

Me ether: methebenol. $C_{18}H_{16}O_3$. MW, 280. Plates from AcOH. M.p. 133–4°. Very sol. $CHCl_3$. *Picrate*: m.p. 106°.

Et ether: $C_{19}H_{18}O_3$. MW, 294. Plates from AcOH. M.p. 104°. Spar. sol. EtOH, ligroin.

Propyl ether: $C_{20}H_{20}O_3$. MW, 308. Plates from AcOH. M.p. 103–5°.

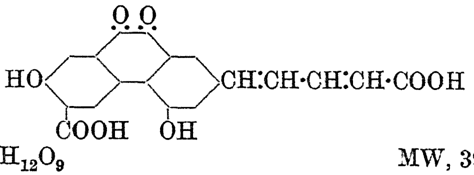
Gulland, Virden, *J. Chem. Soc.*, 1928, 921.
Sieglitz, Koch, *Ber.*, 1925, **58**, 78.
Pschorr, *Ann.*, 1910, **373**, 61.
Freund, Holthof, *Ber.*, 1899, **32**, 184.
Freund, Michaels, *Ber.*, 1897, **30**, 1380.

Theelin.
See Œstrone.

Theelol.
See Œstriol.

Theïne.
See Caffeïne.

Thelephoric Acid



$C_{20}H_{12}O_9$ MW, 396

Isolated from *Thelephora palmata*. Black powder with metallic reflex. Almost insol. in all org. solvents except Py.

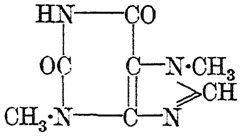
Triacetyl: orange-yellow needles from $PhNO_2$. Decomp. at 330°.

Kögl, Erxleben, Jänecke, *Ann.*, 1930, **482**, 110.

Thenyl Alcohol.
See 2-Aminomethylthiophene.

Thenylamine.
See 2-Hydroxymethylthiophene.

Theobromine (3 : 7-*Dimethylxanthine*, 3 : 7-*dimethyl*-2 : 6-*dihydroxypurine*)



$C_7H_8O_2N_4$ MW, 180

Important alkaloid constituent of cacao beans, kola nuts, etc. Rhombic microcryst. Sublimes at 290°. Insol. ligroin, cold Et_2O, cold $CHCl_3$, C_6H_6. Sol. 1600 parts H_2O at 17°, 3125 parts Et_2O at b.p. $k = 11{\cdot}1 \times 10^{-11}$ at 25° ($4{\cdot}6 \times 10^{-14}$ at 40°). $CrO_3 \longrightarrow$ methylparabanic acid + methylamine. Methylation $\longrightarrow$ caffeine. Faintly basic. Salts decomp. by H_2O. Forms more stable salts with bases. Alkaloid and salts have diuretic action.

Perchlorate: cryst. + $1H_2O$. Decomp. at 271–3°.

Hg salt: darkens at 295–305°. M.p. 310°.

B,HgNO₃: prisms. Does not melt below 300°.

N-*Me*: *see* Caffeïne.

N-*Et*: $C_9H_{12}O_2N_4$. MW, 208. Needles from H_2O. M.p. 164–5°. Sublimes. *B,HAuCl₄*: needles. M.p. 226°.

N-*Propyl*: $C_{10}H_{14}O_2N_4$. MW, 222. Needles from H_2O. M.p. 136°. *B,HAuCl₄*: needles. M.p. 95°.

N-*Butyl*: $C_{11}H_{16}O_2N_4$. MW, 236. Needles from H_2O. M.p. 119°.

N-*Isobutyl*: m.p. 129–30°. *B,HAuCl₄*: needles. M.p. 97°.

Methochloride: colourless columns. M.p. 320–40° decomp. Very sol. H_2O.

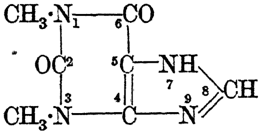
Methochloroaurate: yellow woolly needles. M.p. 265° decomp.

Biltz, Max, *Ann.*, 1921, **423**, 320.
Dubosc, *Chem. Zentr.*, 1932, IV, 956.
Gepner, Kreps, *Chem. Abstracts*, 1947, **41**, 96.

Theocin.

See Theophylline.

Theophylline (*Theocin*, 1 : 3-*dimethylxanthine*, 1 : 3-*dimethyl*-2 : 6-*dihydroxypurine*)



$C_7H_8O_2N_4$ MW, 180

Alkaloid constituent of tea leaves. Needles or plates + $1H_2O$ from hot H_2O. M.p. 264°. Very sol. warm H_2O. Spar. sol. cold EtOH. $k = 16{\cdot}2 \times 10^{-10}$ at 25° ($5{\cdot}46 \times 10^{-14}$ at 40°). Methylation $\longrightarrow$ caffeïne. Alkaloid and metallic salts have diuretic action. Comps. with Na acetate and Na salicylate are important diuretics.

7-N-*Me*: *see* Caffeïne.

7-N-*Propyl*: $C_{10}H_{14}O_2N_4$. MW, 222. *B,HAuCl₄*: yellow needles + $2H_2O$. M.p. 214°.

7-N-*Isopropyl*: *B,HAuCl₄*: yellow needles from EtOH.Aq. M.p. 183°. *B,H₂PtCl₆*: orange-yellow cryst. + $2H_2O$. M.p. 201°.

7-N-*Acetyl*: needles from $CHCl_3$. M.p. 158°.

7-N-*Benzoyl*: needles from EtOH. M.p. 202°.

Yoshitomi, *Chem. Abstracts*, 1925, **19**, 2303.
Biltz, Strufe, *Ann.*, 1914, **404**, 137, 170.
Schwabe, *Arch. pharm.*, 1907, **245**, 312.
Fischer, *Ber.*, 1897, **30**, 553.

Thermopsine

$C_{15}H_{20}ON_2$ MW, 244

Alkaloid isolated from *Thermopsis lanceolata*. Pale yellow cryst. from Me_2CO. M.p. 206–206·5° (203–5°). Sol. H_2O, Et_2O, EtOH, $CHCl_3$. Spar. sol. Me_2CO, pet. ether. $[\alpha]_D^{20}$ − 159·6° in EtOH.

B,HCl: m.p. 247–8° decomp.

B,HI: needles from EtOH. M.p. 294–6°. (306–8° decomp.).

Chloroaurate: m.p. 183–4° decomp.

Chloroplatinate: orange-red prisms. Decomp. at 245–7° (254–6°).

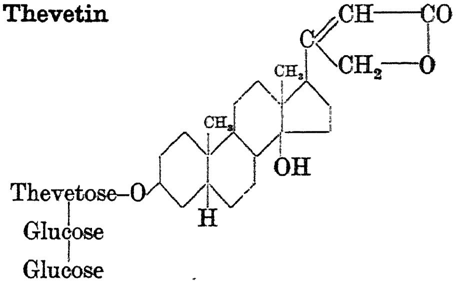
Methiodide: prisms from MeOH. M.p. 241–2° (250°) decomp.

Picrate: yellow prisms from EtOH-Me_2CO. M.p. 229–31° decomp. (208–9°, 262°).

Picrolonate: m.p. 173–5° decomp.

Orechoff, *Chem. Zentr.*, 1935, II, 2215.
Orechoff, Gurewitsch, *Ber.*, 1935, **68**, 820.
Orechoff, Norkina, Gurewitsch, *Ber.*, 1933, **66**, 627.

Thevetin

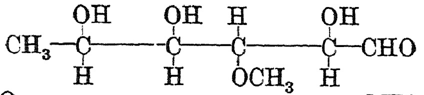


$C_{42}H_{66}O_{18}$ MW, 858

Glycoside from leaves of *Thevetia neriifolia*, Juss., etc. Needles from EtOH or isopropyl alcohol. Softens at 193–4°. M.p. 210°. Sol. EtOH, C_3H_7OH, Py. Spar. sol. H_2O, Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Gives positive Legal test. $[\alpha]_D^{28}$ − 62·5°. Hyd. $\longrightarrow$ glucose + neriifolin.

Tschesche, *Ber.*, 1936, **69**, 2368.
Elderfield, *J. Biol. Chem.*, 1936, **115**, 247.
Chen, Chen, *J. Biol. Chem.*, 1934, **105**, 231.
Helfenberger, Reichstein, *Helv. Chim. Acta*, 1948, **31**, 1470.
Frèrejacque, *Compt. rend.*, 1945, **221**, 645; 1947, **225**, 695.

Thevetose (*Glucomethylose 3-methyl ether*)



$C_7H_{14}O_5$ MW, 178

l-. Cerberose.

Glycosidic constituent of heart poisons occurring in *Thevetia neriifolia*, *Tanghinia venenifera*, and *Cerbera odollam*. Needles from Me_2CO–Et_2O. M.p. 128–30°. $[\alpha]_D^{19}$ − 66° ± 2° in H_2O after 13 minutes ⟶ − 37° ± 2° after 6 hours.

β-*Triacetyl deriv.*: needles from Et_2O–pet. ether. M.p. 118–19°. $[\alpha]_D^{20}$ − 7° in Me_2CO.

Osazone: m.p. 126–30°. $[\alpha]_D$ + 125·5° in EtOH.

d-.

Occurs in a glycoside found in *Adenium honghel*. Cryst. from Me_2CO–Et_2O. M.p. 116°.

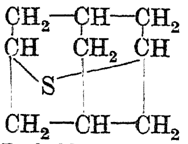
α-*Triacetyl deriv.*: needles from Me_2CO–Et_2O. M.p. 105°. $[\alpha]_D^{20}$ + 122° in Me_2CO.

β-*Triacetyl deriv.*: needles from Me_2CO–Et_2O. M.p. 121°. $[\alpha]_D^{20}$ + 6° in Me_2CO.

Helfenberger, Reichstein, *Helv. Chim. Acta*, 1948, **31**, 1470.

Frèrejacque, *Compt. rend.*, 1950, **230**, 127.

Thia-adamantane



Probable structure

$C_9H_{14}S$ MW, 154

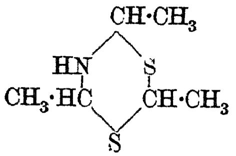
Occurs in Middle East crude oil distillate. Sweet-smelling cryst. M.p. 320° decomp. (sealed tube). B.p. 78°/3 mm. Sublimes. Desulphurization ⟶ bicyclo-1 : 3 : 3-nonane.

Birch, Cullum, Dean, Denyer, *Nature*, 1952, **170**, 629.

Thiacetazone.

See under p-Aminobenzaldehyde.

Thialdine (*Thioacetaldehyde-ammonia, 2 : 4 : 6-trimethyl-5 : 6-dihydro-1 : 3 : 5-dithiazine*)



$C_6H_{13}NS_2$ MW, 163

Cryst. M.p. 43°. Very sol. Et_2O. Sol. EtOH. Spar. sol. H_2O. Volatile in steam. Forms cryst. salts with acids.

B,HCNS: needles. M.p. 132° decomp.

N-*Me*: $C_7H_{15}NS_2$. MW, 177. Needles from EtOH. M.p. 79°. Very sol. Et_2O. Spar. sol. cold EtOH. Insol. cold H_2O. *B,HCNS*: columns from EtOH. M.p. 120°.

Marckwald, *Ber.*, 1886, **19**, 2381.

Thiamin.

See Aneurin.

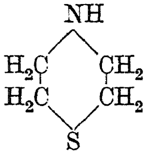
Thianthrene.

See Diphenylene disulphide.

Thiasine.

See Ergothioneine.

1 : 4-Thiazan (*Tetrahydrothiazine, thiomorpholine*)



C_4H_9NS MW, 103

Colourless liq. B.p. 169°. Misc. with H_2O and most org. solvents.

B,HCl: needles from C_6H_6. Softens at 145°. M.p. 160–5° (162°).

Chloroplatinate: does not melt below 250°.

N-*Me*: $C_5H_{11}NS$. MW, 117. B.p. 163–4°. Misc. with H_2O. D_4^{20} 0·9924. n_α^{20} 1·50176. *B,HCl*: m.p. 239°. *Picrate*: yellow needles from EtOH. M.p. 226° decomp.

N-*Et*: $C_6H_{13}NS$. MW, 131. B.p. 184°. Misc. with H_2O. D_4^{20} 0·98854. n_α^{20} 1·50180. *B,HCl*: m.p. 188°. *Chloroplatinate*: decomp. at 222°. *Picrate*: yellow needles from Me_2CO–EtOH. M.p. 185–6°.

N-*Phenyl*: $C_{10}H_{13}NS$. MW, 179. Cryst. from toluene. M.p. 108–11° (33°). B.p. 160–2°/18 mm., 140°/8 mm. *B,HCl*: m.p. 162°. *Picrate*: m.p. 141–2°.

Ethiodide: cryst. from EtOH. M.p. 260°.

Picrate: orange needles from EtOH. M.p. 198° decomp.

Picrolonate: orange prisms from EtOH. M.p. 242° decomp.

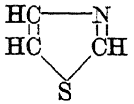
Lawson, Reid, *J. Am. Chem. Soc.*, 1925, **47**, 2830.

Helfrich, Reid, *J. Am. Chem. Soc.*, 1920, **42**, 1226.

Davies, *J. Chem. Soc.*, 1920, **117**, 306.

Clarke, *J. Chem. Soc.*, 1912, **101**, 1586.

Thiazole



C_3H_3NS MW, 85

Liq. B.p. 116·8°. D^{17} 1·1998.

B,HgCl₂: cryst. ppt. Softens at 200°. M.p. 225° decomp.

B,HgCl₂,HCl: needles. M.p. 103–4°.

B_2,H_2PtCl_6: prisms + $2H_2O$. Decomp. at 250°.

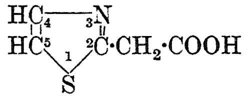
$B,HAuCl_4$: yellow prisms from EtOH. M.p. 258° decomp.

Picrate: needles. Sinters at 150°. M.p. 159–60° decomp.

Gabriel, Bachstez, *Ber.*, 1914, **47**, 3169.

Popp, *Ann.*, 1889, **250**, 275.

Thiazole-2-acetic Acid



$C_5H_5O_2NS$ MW, 143

Needles from aq. sol. of Na salt on acidification. M.p. 109–10°. Very sol. EtOH, Me_2CO, AcOH. Sol. H_2O. Mod. sol. Et_2O, $CHCl_3$. Insol. ligroin.

Me ester: yellow oil. B.p. 95°/14 mm.

Et ester: yellow oil. B.p. 120–2°/15 mm. *Picrate*: golden-yellow needles from EtOH. M.p. 144–6°.

Amide: prisms from EtOH. M.p. 115–16°.

Hydrazide: needles from MeOH. M.p. 116–17°.

Morey, Schenkel, *Helv. Chim. Acta*, 1950, **33**, 405.

Thiazole-4-acetic Acid.

Long colourless needles from EtOH or C_6H_6. M.p. 139°. Sublimes at 60–75°/0·0001 mm.

Et ester: oil. B.p. 160°/71 mm., 122°/15 mm., 111°/2 mm. n_D^{17} 1·5115. *Picrate*: silky pale yellow needles from MeOH. M.p. 112°.

β-*Diethylaminoethyl ester*: pale yellow oil. B.p. 186°/29 mm., 80–90°/0·0001 mm. n_D^{21} 1·5048. Sol. H_2O. *Dipicrate*: cryst. from AcOEt. M.p. 102°.

Amide: plates from dioxan. M.p. 123°.

Nitrile: b.p. 110°/5 mm., 97–8°/2 mm. $n_D^{28.5}$ 1·5391.

Burger, Ullyot, *J. Org. Chem.*, 1947, **12**, 342.

Jones, Robinson, Strachan, *J. Chem. Soc.*, 1946, 87.

Aeberli, Erlenmeyer, *Helv. Chim. Acta*, 1950, **33**, 503.

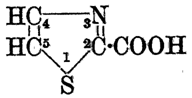
Thiazole-5-acetic Acid.

Needles from aq. sol. of Na salt on acidification with conc. HCl. M.p. 153–4°. Sublimes at 130–40°/12 mm. Sol. EtOH. Mod. sol. H_2O, C_6H_6.

Et ester: golden cryst. from light yellow oil on dist. M.p. 80–1°. B.p. 99–101°/0·7 mm., 59–60°/0·001 mm. *Picrate*: cryst. from EtOH. M.p. 110–11°.

Aeberli, Erlenmeyer, *Helv. Chim. Acta*, 1950, **33**, 503.

Thiazole-2-carboxylic Acid



$C_4H_3O_2NS$ MW, 129

White flocks from aq. sol. of Na salt on acidification with conc. HCl. M.p. 102° under CO_2. Sol. cold EtOH.Aq.

Et ester: needles from 30% EtOH.Aq. M.p. 48°. Sol. hot H_2O, EtOH, Et_2O.

Amide: needles from EtOH.Aq. M.p. 118°.

Erlenmeyer, Marbet, Schenkel, *Helv. Chim. Acta*, 1945, **28**, 924.

Thiazole-4-carboxylic Acid.

Cryst. from amyl alcohol. M.p. 196–7°.

Et ester: oil. B.p. 130–1°/12 mm. Sol. H_2O, EtOH, Et_2O.

Chloride: needles from pet. ether. M.p. 85°.

Amide: cryst. from H_2O. M.p. 150°.

Diethylamide: oil. B.p. 155°/14 mm.

Hydrazide: cryst. from EtOH. M.p. 144°. Very sol. H_2O.

Erlenmeyer, Morel, *Helv. Chim. Acta*, 1945, **28**, 362.

Thiazole-5-carboxylic Acid.

Cryst. from aq. sol. of Na salt on acidification with conc. HCl. M.p. 196–7°.

Me ester: cryst. from pet. ether. M.p. 68–9°.

Et ester: oil.

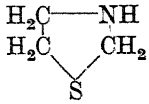
Hydrazide: needles from MeOH. M.p. 157–9°. Turns red in air.

Azide: cryst. from Et_2O. M.p. 103°.

Nitrile: cryst. from Et_2O–pet. ether. M.p. 53°. B.p. 90–3°/15 mm.

Erlenmeyer, Mengisen, Prijs, *Helv. Chim. Acta*, 1947, **30**, 1865.

Thiazolidine (*Tetrahydrothiazole*)



C_3H_7NS MW, 89

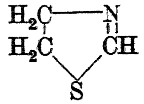
Liq. B.p. 164–5°. D_4^{25} 1·131. n_D^{30} 1·551. pK 6·31. Misc. with H_2O. Volatile in steam. Br in AcOH ⟶ taurine.

B,HCl: cryst. from EtOH. M.p. 180° decomp.

Acetyl: b.p. 83–5°/0·7 mm. *Sulphone*: cryst. from hot EtOH. M.p. 122°.

Ratner, Clarke, *J. Am. Chem. Soc.*, 1937, **59**, 200.

2 : 3-Thiazoline (4 : 5-*Dihydrothiazole*)



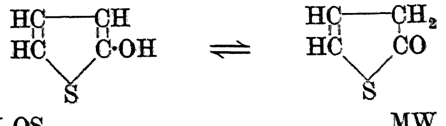
C_3H_5NS MW, 87

B.p. 137·5–138°/747 mm.

Picrate: yellow plates. M.p. 150–1°.

Gabriel, *Ber.*, 1916, **49**, 1111.

2-Thienol (2-*Hydroxythiophene*)



C_4H_4OS MW, 100

Colourless liq. with a non-phenolic odour. F.p. 7–9°. B.p. 217–19°/760°. D_4^{20} 1·255. n_D^{20} 1·5644.

Acetyl: oil. B.p. 96°/25 mm.

Benzoyl: cryst. from EtOH. M.p. 44–5°.

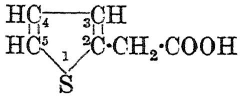
Me ether: liq. B.p. 153–5°.

Hurd, Kreuz, *J. Am. Chem. Soc.*, 1950, **72**, 5543.

Thienone.

See Dithienyl Ketone.

2-Thienylacetic Acid (*Thiophene-2-acetic acid*)



$C_6H_6O_2S$ MW, 142

Cryst. M.p. 76°. Sol. hot H_2O, EtOH, Et_2O.

Me ester: b.p. 115–18°/23 mm.

Et ester: b.p. 124–9°/26 mm.

Nitrile: b.p. 115–20°/22 mm.

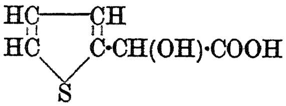
Ernst, *Ber.*, 1886, **19**, 3281.

3-Thienylacetic Acid (*Thiophene-3-acetic acid*).

Cryst. from pet. ether. M.p. 79–80°.

Campaigne, LeSuer, *J. Am. Chem. Soc.*, 1948, **70**, 1555.

2-Thienylglycollic Acid

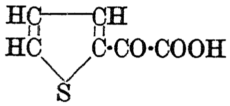


$C_6H_6O_3S$ MW, 158

Needles from C_6H_6. M.p. 115°. Very sol. H_2O, EtOH, Et_2O. Red. ⟶ thienylacetic acid. $MnO_2 + H_2O$ ⟶ thiophene-2-aldehyde.

Ernst, *Ber.*, 1886, **19**, 3281.

2-Thienylglyoxylic Acid



$C_6H_4O_3S$ MW, 156

Cryst. + $1H_2O$. M.p. 58–9°, anhyd. 91·5°. Very sol. H_2O. Very strong acid. Heat ⟶ thiophene-2-aldehyde. Ox. ⟶ thiophene-2-carboxylic acid. AcOH sol. + AcOH–H_2SO_4 ⟶ brown ⟶ green ⟶ violet ⟶ blue col.

Me ester: $C_7H_6O_3S$. MW, 170. M.p. 28·5°. *Oxime*: needles from EtOH.Aq. Softens at 97°. M.p. 104–5°.

Et ester: $C_8H_8O_3S$. MW, 184. Oil with fragrant odour. B.p. 264–5° slight decomp., 115–20°/3 mm. *Oxime*: needles. M.p. 122–3°.

Amide: $C_6H_5O_2NS$. MW, 155. Needles. M.p. 88°. Very sol. EtOH, Et_2O. Insol. H_2O. *Oxime*: needles. M.p. 145–6° decomp. *Acetyl*: prisms. M.p. 85–7° decomp.

Phenylhydrazone: yellow needles from Et_2O. M.p. 164–5° decomp.

Bradley, *Ber.*, 1886, **19**, 2116.

Peter, *Ber.*, 1885, **18**, 537.

Blicke, Tsao, *J. Am. Chem. Soc.*, 1944, **66**, 1645.

Thienylmethyl Alcohol.

See 2-Hydroxymethylthiophene.

Thienylmethylamine.

See 2-Aminomethylthiophene.

Thi-irane.

See Ethylene sulphide.

Thioacetic Acid

$$CH_3{\cdot}CO{\cdot}SH \rightleftharpoons CH_3{\cdot}CS{\cdot}OH$$

C_2H_4OS MW, 76

B.p. 93°. D^{10} 1·075. Very sol. EtOH. Sol. H_2O. $k = 4{\cdot}69 \times 10^{-4}$ at 25°.

Bi salt: prisms. M.p. 85°.

S-*Me ester*: C_3H_6OS. MW, 90. B.p. 95–6°.

O-*Me ester*: pale yellow liq. B.p. 88–91°. D_4^{28} 0·9002. n_D^{28} 1·4212. Insol. H_2O.

S-*Et ester*: C_4H_8OS. MW, 104. B.p. 116–17°. D_4^{28} 0·9755. n_D^{28} 1·4503.

O-*Et ester*: b.p. 105–7°. D_4^{17} 0·8980.

S-*Propyl ester*: $C_5H_{10}OS$. MW, 118. B.p. 135–7°.

O-*Propyl ester*: b.p. 125–30°. D_4^{28} 0·8952. n_D^{28} 1·4283.

S-*Isopropyl ester*: b.p. 124–7°.

O-*Isopropyl ester*: b.p. 119–22°. D_4^{22} 0·8901.

O-*Butyl ester*: $C_6H_{12}OS$. MW, 132. B.p. 146–9°. D_4^{29} 0·8883. n_D^{22} 1·4501, n_D^{28} 1·4196.

O-*Isobutyl ester*: b.p. 135–40°. D_4^{26} 0·8875. n_D^{26} 1·4316.

S-*Phenyl ester*: C_8H_8OS. MW, 152. B.p. 228–30°. D^{23} 1·117. n_D^{22} 1·5706.

O-*Phenyl ester*: b.p. 90–4°/38 mm. D_4^{20} 0·9914.

O-*Benzyl ester*: $C_9H_{10}OS$. MW, 166. B.p. 115–20°/27–9 mm. D_4^{25} 1·0296. n_D^{26} 1·5492.

Anhydride: *see* Diacetyl sulphide.

Amide: thioacetamide. C_2H_5NS. MW, 75. Prisms from Et_2O. M.p. 107·5–108·5° (115°). Sol. H_2O, EtOH. Spar. sol. Et_2O.

Hydrazide: m.p. 59°.

Anilide: thioacetanilide. Needles from H_2O. M.p. 75–6°.

o-*Toluidide*: needles from ligroin. M.p. 91–2°.

m-*Toluidide*: needles. M.p. 42–3°.

p-*Toluidide*: prisms from EtOH.Aq. M.p. 129·5–130·5°.

o-*Anisidide*: yellow plates. M.p. 52–3°.

p-*Anisidide*: needles. M.p. 114°.

Baker, Reid, *J. Am. Chem. Soc.*, 1929, **51**, 1568.

Sakurada, *Brit. Chem. Abstracts*, 1927, 133.

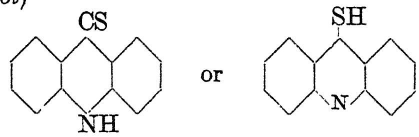
Clarke, Hartman, *J. Am. Chem. Soc.*, 1924, **46**, 1732.

Delépine, *Compt. rend.*, 1911, **153**, 281.

Schoff, *Ber.*, 1895, **28**, 1205.

Thioacridol.
See Thioacridone.

Thioacridone (9-*Mercaptoacridine, thioacridol*)



$C_{13}H_9NS$ MW, 211

Brownish-yellow needles + $1H_2O$ from 2% NaOH. M.p. 275°. Loses H_2O at 120° over H_2SO_4. Sol. Me_2CO. Spar. sol. other org. solvents. Sol. conc. acids, alkalis and NH_4OH. Insol. alkali carbonates. Salts are unstable and decomp. by H_2O.

S-*Me*: $C_{14}H_{11}NS$. MW, 225. Greenish-yellow needles from EtOH. M.p. 113–14°. *Hydrochloride*: yellow needles. M.p. 198°. *Sulphate*: m.p. 156–7°. *Nitrate*: m.p. 117–18°. *Picrate*: needles from EtOH–$CHCl_3$. M.p. 205°.

S-*Et*: $C_{15}H_{13}NS$. MW, 239. Yellow needles from pet. ether. M.p. 65°. *Picrate*: yellow needles from $CHCl_3$–EtOH. M.p. 182–3°.

S-2 : 4-*Dinitrophenyl*: yellow needles from xylene. M.p. 290° decomp. *Picrate*: yellow needles. M.p. 226° decomp.

S-2 : 4 : 6-*Trinitrophenyl*: thioacridol picryl ether. Red needles from $CHCl_3$. M.p. 233° decomp.

S-*Benzyl*: needles from EtOH.Aq. M.p. 109°. *Hydrochloride*: yellow leaflets. Decomp. at 140–1°. *Sulphate*: m.p. 179–80° decomp. *Nitrate*: yellow leaflets. M.p. 106–7° decomp. *Picrate*: yellowish-brown needles from EtOH. M.p. 189–90°.

S-o-*Nitrobenzyl*: yellow leaflets. M.p. 129–30°. *Picrate*: yellow cryst. M.p. 190–1°.

S-p-*Nitrobenzyl*: yellow cryst. from Me_2CO. M.p. 152°. *Picrate*: m.p. 204°.

S-*Benzoyl*: yellow leaflets from EtOH–$CHCl_3$. M.p. 209°. *Picrate*: greenish-yellow needles from EtOH–$CHCl_3$. M.p. 190°.

N-*Me*: red needles from xylene. M.p. 263°.

N-*Et*: red needles from xylene. M.p. 218°.

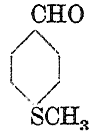
N-*Phenyl*: $C_{19}H_{13}NS$. MW, 287. Red cryst. from AcOH. M.p. 227–8°. Sol. Me_2CO, $CHCl_3$, C_6H_6. Mod. sol. EtOH, AcOH.

Edinger, *J. prakt. Chem.*, 1903, **68**, 88.
Edinger, Arnold, *J. prakt. Chem.*, 1901, **64**, 196, 487.
Kalle, D.R.P., 120,586, (*Chem. Zentr.*, 1901, I, 1254).
Schönberg, Schütz, Nickel, *Ber.*, 1928, **61**, 1383.
Asquith, Hammick, Williams, *J. Chem. Soc.*, 1948, 1181.

Thioamyl Alcohol.
See Amyl Mercaptan.

Thioaniline.
See 4 : 4′-Diaminodiphenyl sulphide.

Thioanisaldehyde (p-*Methylmercaptobenzaldehyde*)



C_8H_8OS MW, 152

Yellowish plates from ligroin. M.p. 78°. B.p. 273°. Sol. common org. solvents.

Phenylhydrazone: m.p. 138° (136°).

Friedländer, Lenk, *Ber.*, 1912, **45**, 2089.
Mitra, *Chem. Abstracts*, 1933, **27**, 3923.

Thioanisidine.
See under Aminothiophenol.

Thioanisole (*Methyl phenyl sulphide*)

$C_6H_5·SCH_3$

C_7H_8S MW, 124

B.p. 187–8°, 58–60°/6 mm. D_4^{25} 1·0533. n_D^{25} 1·5832.

Knapp, *Monatsh.*, 1930, **56**, 68 (*Footnote*).
Suter, Hansen, *J. Am. Chem. Soc.*, 1932, **54**, 4101.
Brand, Kranz, *J. prakt. Chem.*, 1927, **115**, 143.

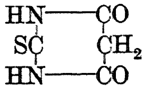
Thioanisole-ω-carboxylic Acid.
See Phenylthioglycollic Acid.

Thioanthrol.
See Mercaptoanthracene.

Thioargyrium.
See under Dithiosalicylic Acid.

Thioaspirin.
See under Thiosalicylic Acid.

Thiobarbituric Acid (*Malonylthiourea*)



$C_4H_4O_2N_2S$ MW, 144

Plates from H_2O. M.p. 235° decomp. (rapid heat.).

Michael, *J. prakt. Chem.*, 1894, **49**, 38.
Harwood, *Chem. Abstracts*, 1933, **27**, 1676.

Thiobenzamide.
See under Thiobenzoic Acid.

Thiobenzanilide.
See under Thiobenzoic Acid.

Thiobenzhydrol (*Benzhydryl mercaptan, α-mercaptodiphenylmethane*)

$C_6H_5·CH(SH)·C_6H_5$

$C_{13}H_{12}S$ MW, 200

Yellow oil. B.p. 128–30°/1·2 mm.

Staudinger, Siegwart, *Ber.*, 1916, **49**, 1920.

Thiobenzilic Acid (*α-Mercaptodiphenylacetic acid*)

$(C_6H_5)_2C(SH)·COOH$

$C_{14}H_{12}O_2S$ MW, 244

Plates from AcOH. M.p. 147·5–149°. Sol. EtOH, Et_2O, Me_2CO, AcOH, C_6H_6, boiling MeOH. Very spar. sol. boiling H_2O.

Bettschart, Bistrzycki, *Helv. Chim. Acta*, 1919, **2**, 127.

Thiobenzoic Acid

C_6H_5·CO·SH or C_6H_5·CS·OH

C_7H_6OS MW, 138

Yellow oil. Solidifies in ice and then melts about 24°. Very sol. EtOH, Et_2O, CS_2. Insol. H_2O. Volatile in steam. Decomp. on dist.

NH_4 salt: cryst. M.p. 118°. Sol. H_2O.

O-*Me ester*: C_8H_8OS. MW, 152. Yellow liq. B.p. 110–12°/10 mm. Insol. H_2O. Fumes in air.

S-*Me ester*: b.p. 231–2°, 134°/25 mm. D_{25}^{25} 1·1381.

S-*Et ester*: $C_9H_{10}OS$. MW, 166. B.p. 252–3°, 146°/31 mm. D_{25}^{25} 1·0977.

S-*Propyl ester*: $C_{10}H_{12}OS$. MW, 180. B.p. 251–5°, 144°/13 mm. D_{25}^{25} 1·0724.

S-*Butyl ester*: $C_{11}H_{14}OS$. MW, 194. B.p. 160°/23 mm. D_{25}^{25} 1·0514.

S-sec.-n-*Butyl ester*: b.p. 151°/23 mm. D_{25}^{25} 1·0488.

S-*Isobutyl ester*: b.p. 150°/20 mm. D_{25}^{25} 1·0457.

S-tert.-*Butyl ester*: b.p. 110°/28 mm. D_{25}^{25} 1·0468.

S-*Phenyl ester*: $C_{13}H_{10}OS$. MW, 214. Needles from EtOH or C_6H_6. M.p. 56°. $n_D^{56·5}$ 1·6231. Sol. EtOH, Et_2O, $CHCl_3$, CS_2. KOH ⟶ thiophenol + benzoic acid.

S-p-*Tolyl ester*: cryst. from EtOH. M.p. 75°.

S-*Benzyl ester*: cryst. from EtOH. M.p. 39·5°.

Amide: thiobenzamide. C_7H_7NS. MW, 137. Needles. M.p. 115–16°. N-*Benzoyl*: red cryst. M.p. 117°.

Methylamide: C_8H_9NS. MW, 151. Yellow needles from EtOH. M.p. 79°.

Anilide: thiobenzanilide. Yellow plates or prisms. M.p. 101·5–102° (96°).

N-*Me-anilide*: m.p. 63° (59°). B.p. 331–2°.

o-*Toluidide*: m.p. 85–6°.

p-*Toluidide*: yellow needles from EtOH.Aq. M.p. 128·5–129·5°.

Piperidide: yellow plates from MeOH.Aq. M.p. 63–4°. B.p. 205°/12 mm.

Anhydride: *see* Dibenzoyl sulphide.

Engelhardt, Latschinow, Malyschew, *Z. Chem.*, 1868, 354.
Kym, *Ber.*, 1899, **32**, 3533 (*Note*).
Hantzsch, Scharf, *Ber.*, 1913, **46**, 3584.
Gabriel, Heymann, *Ber.*, 1890, **23**, 158.

Thiobenzophenone (*Diphenyl thioether*)

C_6H_5·CS·C_6H_5

$C_{13}H_{10}S$ MW, 198

Blue needles from pet. ether. M.p. 53–4°. B.p. 174°/14 mm. Very sol. $CHCl_3$, C_6H_6. Spar. sol. pet. ether, cold EtOH. Standing in air ⟶ benzophenone.

Staudinger, Freudenberger, *Organic Syntheses*, 1931, XI, 94.

Thiobenzyl Alcohol.
See Benzyl Mercaptan.

Thiobiuret

H_2N·CO·NH·CS·NH_2 (5 4 3 2 1)

$C_2H_5ON_3S$ MW, 119

Needles + $1H_2O$ from H_2O. M.p. anhyd. 186°. Sol. EtOH, AcOH. Mod. sol. H_2O. Insol. CS_2, ligroin, C_6H_6. Sol. alkalis.

5-*Phenyl*: cryst. from EtOH. M.p. 186°.

1 : 5-*Diphenyl*: m.p. 161°.

Wunderlich, *Ber.*, 1886, **19**, 452.
Hecht, *Ber.*, 1892, **25**, 749.

Thiobutyric Acid

CH_3·CH_2·CH_2·CO·SH or CH_3·CH_2·CH_2·CS·OH

C_4H_8OS MW, 104

B.p. 130°. Sol. EtOH. Spar. sol. H_2O.

S-*Phenyl ester*: $C_{10}H_{12}OS$. MW, 180. Yellow liq. B.p. 210–12°/20 mm. Volatile in steam.

Anilide: needles from AcOH.Aq. M.p. 32–3°.

Ulrich, *Ann.*, 1859, **109**, 280.

Thiocarbamic Acid

H_2N·CO·SH or H_2N·CS·OH

CH_3ONS MW, 77

Known only in form of its salts and esters.

NH_4 salt: colourless cryst. Very sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. Dry salt rapidly turns yellow in air ⟶ $(NH_4)_2S$.

S-*Me ester*: C_2H_5ONS. MW, 91. Prisms from Et_2O. M.p. 107–8° (95–8°). N-*Acetyl*: needles from C_6H_6. M.p. 145·5–146°.

O-*Me ester*: m.p. 43°. N-*Acetyl*: cryst. from pet. ether. M.p. 79–80°.

Et ester: *see* Thiourethane.

O-*Propyl ester*: C_4H_9ONS. MW, 119. Cryst. M.p. 35°.

S-*Isopropyl ester*: plates. M.p. 125°.

S-*Isobutyl ester*: $C_5H_{11}ONS$. MW, 133. Plates from H_2O. M.p. 102–3°. Volatile in steam.

O-*Isobutyl ester*: plates from H_2O. M.p. 51–3° (36°).

S-*Phenyl ester*: C_7H_7ONS. MW, 153. Plates from C_6H_6 or H_2O. M.p. 96–8°.

O-*Phenyl ester*: needles from EtOH. M.p. 132–132·5°.

S-*Benzyl ester*: C_8H_9ONS. MW, 167. Plates from C_6H_6. M.p. 125°.

Amide: *see* Thiourea.

Wheeler, Barnes, *Am. Chem. J.*, 1899, **22**, 146.
Kretzschmar, *J. prakt. Chem.*, 1873, **7**, 474.
Salomon, *J. prakt. Chem.*, 1873, **8**, 115.

Thiocarbamide.
See Thiourea.

Thiocarbanilic Acid.
See Phenylthiocarbamic Acid.

Thiocarbanilide (sym.-*Diphenylthiourea*)

$C_6H_5·NH·CS·NH·C_6H_5$

$C_{13}H_{12}N_2S$ MW, 228

Leaflets from EtOH. M.p. 154–5° (150·5°). Sol. EtOH, Et_2O. Mod. sol. H_2O. Insol. CS_2. Sol. alkalis, re-ppd. by acids. Triboluminescent. Very bitter taste.

$B,HgCl_2$: m.p. 85°.
B_2,HgI_2: m.p. 183°.
B,HgI_2: yellow prisms. M.p. 139°.
$B_2,AuCl_3$: prisms. M.p. 194°.
$B_2,CuBr_2$: prisms. M.p. 187°.
$B_2,CdBr_2$: m.p. 140°.
$B_2,ZnCl_2$: prisms. M.p. 172°.
$B_2,SnCl_4$: prisms. M.p. 260°.

Fry, *J. Am. Chem. Soc.*, 1913, **35**, 1541.
Drozdov, *Chem. Abstracts*, 1932, **26**, 5293.
Mistry, Guha, *Chem. Abstracts*, 1931, **25**, 1504.
Silesia Verein, D.R.P., 559,814, (*Chem. Zentr.*, 1933, I, 2463).
Rubber Service Labs., U.S.P., 1,688,707, (*Chem. Abstracts*, 1929, **23**, 156).

Thiocarbazide (*Thiocarbohydrazide, dihydrazide of thiocarbonic acid*)

$H_2N·NH·CS·NH·NH_2$
5 4 3 2 1

CH_6N_4S MW, 106

Cryst. from H_2O. Decomp. at 170°.
1 : 5-*Diacetyl*: hygroscopic cryst. M.p. 204–5°.
1-*Phenyl*: $C_7H_{10}N_4S$. MW, 182. Tables from EtOH. M.p. 149–50°. Sol. EtOH, AcOH, Py, alkalis. Spar. sol. Me_2CO, $CHCl_3$, C_6H_6, HCl. *B,HCl*: plates. M.p. 181°.
1 : 5-*Diphenyl*: $C_{13}H_{14}N_4S$. MW, 258. M.p. 156–8°.
1-*Me*-1-*Phenyl*: $C_8H_{12}N_4S$. MW, 196. Prisms from EtOH. M.p. 228–9° decomp.

Guha, Roy-Choudhury, *J. Indian Chem. Soc.*, 1928, **5**, 149.
Guha, De, *J. Chem. Soc.*, 1924, 1215.
Billman, Cleland, *Organic Syntheses*, 1945, XXV, 38.

Thiocarbonic Acid

HO·CO·SH or HO·CS·OH

CH_2O_2S MW, 78

Does not exist in free state.
O : S-*Di-Me ester*: $C_3H_6O_2S$. MW, 106. Oil with ethereal odour. B.p. 120–1°. $D_4^{21·5}$ 1·1203. n_D^{23} 1·4524.
O-*Di-Me ester*: b.p. 119–20°. D_4^2 1·1028. n_D^{24} 1·4596.
O-S-*Di-Et ester*: $C_5H_{10}O_2S$. MW, 134. B.p. 156°. D^{18} 1·0285.
O-*Di-Et ester*: b.p. 158–9°. $D_4^{17·5}$ 1·0267. $n_D^{17·5}$ 1·4601.
O : S-*Diphenyl ester*: $C_{13}H_{10}O_2S$. MW, 230. Needles from EtOH. M.p. 56°.
O-*Diphenyl ester*: plates from EtOH. M.p. 106°. Sol. most ord. org. solvents. Insol. H_2O.
Anhydride: *see* Carbonyl sulphide.
Amide: *see* Thiocarbamic Acid.
Chloride: *see* Thiocarbonyl chloride.
Hydrazide: *see* Thiocarbazide.
Nitrile: *see* Thiocyanic acid.
Anil: *see* Phenyl isothiocyanate.

Delépine, *Ann. chim.*, 1912, **25**, 547; *Compt. rend.*, 1910, **150**, 878; *Bull. soc. chim.*, 1910, **7**, 409, 727.

Thiocarbonyl chloride (*Thiophosgene*)

$CSCl_2$

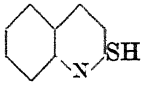
CCl_2S MW, 115

Red liq. with acrid odour. B.p 73–6°. Fumes in air. D^{15} 1·5085. n_D^{20} 1·5442. When pure is not decomp. by light. Slowly decomp. by cold H_2O, rapidly with hot EtOH or NaOEt ⟶ esters.

Dyson, *Organic Syntheses*, 1926, VI, 86.

Thiocarbonyl tetrachloride.
See Perchloromethyl Mercaptan.

Thiocarbostyril (2-*Mercaptoquinoline*, 2-*quinolyl mercaptan*)

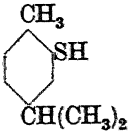


C_9H_7NS MW, 161

Yellow plates from EtOH.Aq. M.p. 175°. Very sol. hot EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Insol. cold H_2O. Sol. acids, alkalis.
S-*Me*: $C_{10}H_9NS$. MW, 175. M.p. 55°. B.p. 182–3°/22 mm. *Methiodide*: yellow cryst. from EtOH. M.p. 189°.
N-*Me*: greenish-yellow needles or prisms from EtOH. M.p. 118°. Spar. sol. hot H_2O.
S-*Et*: $C_{11}H_{11}NS$. MW, 189. Liq. B.p. 177–8°/26 mm. Decomp. on dist. at atm. press. *B,HI*: yellow cryst. from EtOH–Et_2O. M.p. 154°. *B_2,H_2PtCl_6*: cryst. + H_2O. M.p. 190° decomp.

Fischer, *Ber.*, 1899, **32**, 1305.

Thiocarvacrol (2-*Mercapto*-p-*cymene*, 2-*methyl*-5-*isopropylthiophenol*)

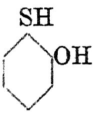


$C_{10}H_{14}S$ MW, 166

Colourless liq. with aromatic odour. B.p. 235–6°. $D^{17·5}$ 0·9975. Misc. with EtOH. Insol. H_2O.
Hg salt: needles. M.p. 109°.
S-*Me*: $C_{11}H_{16}S$. MW, 180. Liq. with unpleasant odour. B.p. 244°.

Kekulé, Fleischer, *Ber.*, 1873, **6**, 1088.

Thiocatechol (*2-Hydroxythiophenol, o-hydroxyphenyl mercaptan*)



C_6H_6OS MW, 126

Oil with strong odour. M.p. 5–6°. B.p. 216–17°/750·7 mm. D_0^0 1·2373. Sol. Et_2O. Spar. sol. H_2O. Readily oxidises in alk. sol. to disulphide.

O-Me ether: *o*-methoxythiophenol, *o*-mercaptoanisole, thioguiacol. C_7H_8OS. MW, 140. Liq. B.p. 218–19°.

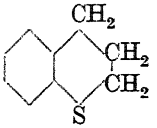
O-Et ether: *o*-mercaptophenetole. $C_8H_{10}OS$. MW, 154. Liq. B.p. 226–7°.

Di-Me ether: *o*-methoxythioanisole. $C_8H_{10}OS$. MW, 154. Liq. B.p. 237°.

Di-Et ether: *o*-ethoxythiophenetole. $C_{10}H_{14}OS$. MW, 182. Liq. B.p. 248–50°.

Friedländer, Mauthner, *Chem. Zentr.*, 1904, II, 1176.
Gattermann, *Ber.*, 1899, **32**, 1147.
Haitinger, *Monatsh.*, 1883, **4**, 170.

Thiochroman (*Dihydrobenzthiopyran*)



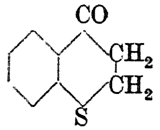
$C_9H_{10}S$ MW, 150

Yellow liq. B.p. 128–30°/15 mm. Volatile in steam. $KMnO_4 \longrightarrow$ thiochroman-S-dioxide.

S-Dioxide: $C_9H_{10}O_2S$. MW, 182. Cryst. from hot H_2O. M.p. 88·5°. Sol. H_2O. Very sol. Et_2O.

v. Braun, *Ber.*, 1910, **43**, 3225.

Thiochromanone



C_9H_8OS MW, 164

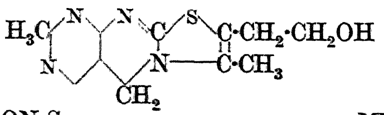
Colourless leaflets with mint-like odour from pet. ether. M.p. 29–30°. B.p. 154°/12 mm. $H_2SO_4 \longrightarrow$ red col.

Oxime: plates from EtOH–petrol. M.p. 98–100°.

Semicarbazone: needles from EtOH. M.p. 219–20°.

Krollpfeiffer, Schultze, *Ber.*, 1923, **56**, 1822.

Thiochrome

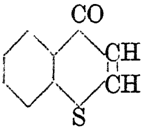


$C_{12}H_{14}ON_4S$ MW, 262

The colouring matter of yeast. Yellow prisms from $CHCl_3$. M.p. 227–8°. Sol. MeOH. Mod. sol. H_2O. Spar. sol. EtOH, Et_2O, Me_2CO, $CHCl_3$. Gives blue fluor. in neutral or alk. sol. Sublimes in high vacuum. Has no physiological activity.

Todd, Bergel, *J. Chem. Soc.*, 1936, 1560.
Todd, Bergel, Fraenkel-Conrat, Jacob, *J. Chem. Soc.*, 1936, 1601.
Kuhn, Vetter, *Ber.*, 1935, **68**, 2375.
Barger, Bergel, Todd, *ibid.*, 2257.
Kuhn, Wagner-Jauregg, Klaveren, Vetter, *Z. physiol. Chem.*, 1935, **234**, 196.
Sykes, Todd, *J. Chem. Soc.*, 1951, 534.

Thiochromone (*1 : 4-Benzthiopyrone*)



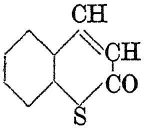
C_9H_6OS MW, 162

Colourless needles from ligroin. M.p. 78°.

Arndt *et al.*, *Ber.*, 1925, **58**, 1620.

Thiocoumarandione.
See Thionaphthenequinone.

1 - Thiocoumarin (*o - Mercaptocinnamic lactam*)



C_9H_6OS MW, 162

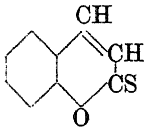
Needles from ligroin. M.p. 80–80·5°. Spar. sol. H_2O. Volatile in steam.

Phenylhydrazone: yellow plates from EtOH. M.p. 140°.

Picrate: m.p. 148°.

Simonis, Elias, *Ber.*, 1916, **49**, 765.

2-Thiocoumarin



C_9H_6OS MW, 162

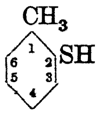
Parent compound not described.

1 : 3 : 5-Trinitrobenzene add. comp.: cryst. from EtOH. M.p. 87°.

Kent, McNeil, Cowper, *J. Chem. Soc.*, 1939, 1858.

Thiocoumarone.
See Thionaphthene.

***o*-Thiocresol** (*o-Mercaptotoluene*)



C_7H_8S MW, 124

Plates. M.p. 15°. B.p. 194·3°, 124·7°/100 mm., 106°/50 mm. Sol. EtOH. Insol. H_2O. Volatile in steam. Ox. ⟶ di-*o*-tolyl disulphide. H_2SO_4 ⟶ blue col.

S-*Et*: $C_9H_{12}S$. MW, 152. Liq. B.p. 120°.

S-*Phenyl*: *see* Phenyl *o*-tolyl sulphide.

S-o-*Tolyl*: *see* Di-*o*-tolyl sulphide.

Hübner, Post, *Ann.*, 1873, **169**, 30.

***m*-Thiocresol** (m-*Mercaptotoluene*).

Liq. B.p. 195·4°, 126°/100 mm., 107·5°/50 mm. Sol. EtOH. Insol. H_2O. Volatile in steam. D_4^0 1·06251. Ox. ⟶ di-*m*-tolyl disulphide.

S-*Phenyl*: *see* Phenyl *m*-tolyl sulphide.

S-m-*Tolyl*: *see* Di-*m*-tolyl sulphide.

Hübner, Post, *Ann.*, 1873, **169**, 51.
Tarbell, Fukushima, *Organic Syntheses*, 1947, XXVII, 81.

***p*-Thiocresol** (p-*Mercaptotoluene*).

Plates from EtOH.Aq. or Et_2O. M.p. 43–4°. B.p. 195°, 124·9°/100 mm., 71·4°/10·6 mm. Sol. EtOH, Et_2O. H_2SO_4 ⟶ blue col. Ox. ⟶ di-*p*-tolyl disulphide. Conc. H_2SO_4 ⟶ 2 : 6-dimethylthianthrene. Produces eczema on skin.

S-*Me*: *see* Methyl *p*-tolyl sulphide.

S-*Et*: $C_9H_{12}S$. MW, 152. B.p. 220–1°, 105°/15 mm. $D^{17·5}$ 1·0016.

S-*Isopropyl*: $C_{10}H_{14}S$. MW, 166. Oil. B.p. 228°, 110°/14 mm.

S-*Phenyl*: *see* Phenyl *p*-tolyl sulphide.

S-p-*Tolyl*: *see* Di-*p*-tolyl sulphide.

S-*Chloroacetyl*: m.p. 40°.

Fischer, *Ber.*, 1915, **48**, 96, 100.
Bourgeois, *Rec. trav. chim.*, 1899, **18**, 437.

Thiocresol-carboxylic Acid.
See Mercaptotoluic Acid.

Thio-*p*-cresotinic Acid.
See 4-Mercapto-*m*-toluic Acid.

Thioctic Acid.
See α-Lipoic Acid.

Thiocyanic Acid

HS·C:N

CHNS MW, 59

Gas. Forms colourless cryst. on cooling which rapidly melt at 5° to yellow liq. Sol. H_2O, EtOH, Et_2O in all proportions. Dil. aq. sols. are stable.

Me ester: *see* Methyl thiocyanate.

Et ester: *see* Ethyl thiocyanate.

Propyl ester: C_4H_7NS. MW, 101. Liq. with unpleasant odour. B.p. 163°.

Isopropyl ester: *see* Isopropyl thiocyanate.

Butyl ester: C_5H_9NS. MW, 115. B.p. 184·5–185·5°/743 mm. D_4^{25} 0·9563.

Isobutyl ester: *see* Isobutyl thiocyanate.

Amyl ester: $C_6H_{11}NS$. MW, 129. B.p. 90–1°/16 mm. D_4^{25} 0·9412. n_D^{25} 1·462.

Isoamyl ester: yellow oil. B.p. 197°.

Allyl ester: b.p. 161°. D^0 1·071.

Phenyl ester: C_7H_5NS. MW, 135. Liq. B.p. 231°. $D^{17·5}$ 1·155.

Benzyl ester: *see* Benzyl thiocyanate.

Allen, *J. Am. Chem. Soc.*, 1935, **57**, 198.
Shriner, *Organic Syntheses*, 1931, XI, 92.
Dienske, *Rec. trav. chim.*, 1927, **46**, 154.
Kaufmann, Oehring, *Ber.*, 1926, **59**, 187.

Thiodiacetic Acid.
See Thiodiglycollic Acid.

Thiodiethylamine.
See 2 : 2′-Diaminodiethyl sulphide.

Thiodiglycol (*Dihydroxydiethyl sulphide*)

HO·CH$_2$·CH$_2$·S·CH$_2$·CH$_2$OH

$C_4H_{10}O_2S$ MW, 122

Syrup. B.p. 164–6°/20 mm. n_D^{26} 1·4628. Misc. in all proportions with H_2O. Combines with $CaCl_2$.

Diphenyl ether: $C_{16}H_{18}O_2S$. MW, 274. Needles from EtOH. M.p. 42°.

Di-1-naphthyl ether: $C_{24}H_{22}O_2S$. MW, 374. Light brown cryst. M.p. 94·5°. Spar. sol. EtOH.

Di-2-naphthyl ether: cryst. M.p. 129°. Spar. sol. EtOH.

Monoacetyl: b.p. 120–1°/34 mm. n_D^{26} 1·4728.

Diacetyl: b.p. 118°/2 mm. D^{35} 1·1270. n_D^{25} 1·4675.

Dibenzoyl: cryst. from EtOH. M.p. 65°.

Di-p-nitrobenzoyl: powder. M.p. 107·7°.

Di-p-aminobenzoyl: powder. M.p. 184·5°.

Nenitzescu, Scărlătescu, *Ber.*, 1935, **68**, 588.
Faber, Miller, *Organic Syntheses*, 1932, XII, 68.
Helfrich, Reid, *J. Am. Chem. Soc.*, 1920, **42**, 1208.
Szabo, Stiller, *J. Am. Chem. Soc.*, 1948, **70**, 3667.

Thiodiglycollic Acid (*Dimethyl sulphide dicarboxylic acid, dicarboxydimethyl sulphide, thiodiacetic acid*)

HOOC·CH$_2$·S·CH$_2$·COOH

$C_4H_6O_4S$ MW, 150

Cryst. from AcOEt–C_6H_6. M.p. 129°. Sol. 2·37 parts H_2O at 18°. Very sol. EtOH. $k = 6–7·5 \times 10^{-4}$ at 25°. Ox. ⟶ dimethyl sulphone dicarboxylic acid. Red. ⟶ acetic acid. H_2O_2 ⟶ thionyldiglycollic acid.

Di-Me ester: $C_6H_{10}O_4S$. MW, 178. B.p. 135°/11 mm.

Di-Et ester: $C_8H_{14}O_4S$. MW, 206. B.p. 267–8°.

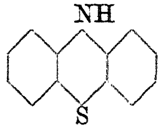
Dibutyl ester: b.p. 131°/2 mm.

Monoamide: $C_4H_7O_3NS$. MW, 149. Prisms. M.p. 125°. Spar. sol. cold H_2O. Sol. hot H_2O. Heat ⟶ imide. o-*Toluidide*: needles from EtOH.Aq. M.p. 150–1°. m-*Toluidide*: cryst. from EtOH.Aq. M.p. 97–8°. p-*Toluidide*: plates from EtOH.Aq. M.p. 148–9°.

Anhydride: $C_4H_4O_3S$. MW, 132. Needles from $CHCl_3$. M.p. 102°. B.p. 158°/10 mm. Very sol. hot $CHCl_3$. Spar. sol. hot Et_2O.
Nitrile: $C_4H_4N_2S$. MW, 112. Plates from MeOH. M.p. 45·5–46·5°.
Monoanilide: cryst. from H_2O. M.p. 103°.
Dianilide: needles from EtOH. M.p. 168°.

Beckurts, Frerichs, *J. prakt. Chem.*, 1906, **74**, 50.
Lovén, *Ber.*, 1894, **27**, 3059.
Barkenbus, Landis, *J. Am. Chem. Soc.*, 1948, **70**, 684.

Thiodiphenylamine (*Phenthiazine, phenothiazine*)

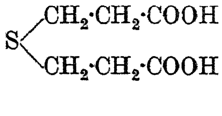


$C_{12}H_9NS$ MW, 199

Yellow plates from EtOH. M.p. 182°. B.p. 371°, 290°/40 mm. Sol. Et_2O, C_6H_6. Spar. sol. cold EtOH, ligroin. Sublimes. Alc. $FeCl_3$ ⟶ green col. Heat with Zn ⟶ diphenylamine. Boil sol. with Cu ⟶ carbazole.
N-*Me*: $C_{13}H_{11}NS$. MW, 213. Exists in two forms. (α-) Long prisms from EtOH. M.p. 99·3°. B.p. 360–5°. Sol. C_6H_6. Mod. sol. Et_2O. Spar. sol. cold EtOH, AcOH. H_2SO_4 ⟶ reddish-brown sol. (β-) Yellow needles from EtOH–C_6H_6. M.p. 78–9°. Sol. warm C_6H_6. Spar. sol. hot EtOH. Insol. H_2O.
N-*Et*: $C_{14}H_{13}NS$. MW, 227. Prisms from EtOH. M.p. 102°.
N-*Phenyl*: $C_{18}H_{13}NS$. MW, 275. Cryst. from EtOH. M.p. 89–90°. B.p. 187–95°/1 mm.
N-*Benzyl*: $C_{19}H_{15}NS$. MW, 289. Cryst. from EtOH. M.p. 130°.
N-*Acetyl*: prisms from EtOH. M.p. 197–197·5°.
N-p-*Toluenesulphonyl*: m.p. 155–6°.
S-*Dioxide*: *see* Diphenylamine sulphone.

Kehrmann, Dardel, *Ber.*, 1922, **55**, 2349.
Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 11.
Holzmann, *Ber.*, 1888, **21**, 2065.
Bernsthen, *Ann.*, 1885, **230**, 88.

Thiodipropionic Acid (*Diethyl sulphide 2 : 2′-dicarboxylic acid*)

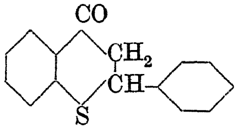


$C_6H_{10}O_4S$ MW, 178

M.p. 129–30°. Anti-oxidant.
Et ester: b.p. 174°/15 mm. D_4^{20} 1·1034. n_D^{20} 1·4694.
Diamide: plates from EtOH. M.p. 177–178·5°.
Dianilide: plates from toluene. M.p. 163–5°.

Bennett, Scorah, *J. Chem. Soc.*, 1927, 196.
American Cyanamid Co., B.P. 571,628, (*Chem. Abstracts*, 1947, **41**, 3119).
Gresham *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 999.
B.F. Goodrich Co., B.P. 618,390, (*Chem. Abstracts*, 1949, **43**, 5795).

Thiododecyl Alcohol.
See Dodecyl Mercaptan.
Thioethanolamine.
See 2-Mercaptoethylamine.
Thioethyl Alcohol.
See Ethyl Mercaptan.
Thioethylamine.
See 2 : 2′-Diaminodiethyl sulphide.
Thioethylene Glycol.
See Ethylene Thioglycol.
Thioflavanone

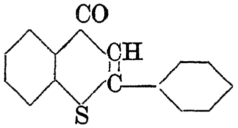


$C_{15}H_{12}OS$ MW, 240

Colourless needles from EtOH or pet. ether–CS_2. M.p. 55–6°. Sol. Et_2O, C_6H_6. Sol. H_2SO_4 ⟶ red col. Insol. alkalis.

Arndt, *Ber.*, 1923, **56**, 1274.

Thioflavone



$C_{15}H_{10}OS$ MW, 238

Needles from EtOH. M.p. 129–30° (125°). Alc. I ⟶ blue col. H_2SO_4 ⟶ yellow col. Forms salts readily hyd. by H_2O.

Arndt *et al.*, *Ber.*, 1925, **58**, 1620.
Ruhemann, *Ber.*, 1913, **46**, 2197.

Thioformic Acid

H·CO·SH

CH_2OS MW, 62

Unstable liq.
S-*Benzyl ester*: b.p. 109–11°/26 mm.
Amide: thioformamide. CH_3NS. MW, 61. Prisms from AcOEt or EtO–pet. ether. M.p. 28–9°. Very sol. EtOH, Me_2CO, AcOEt. Sol. Et_2O. Insol. CS_2, ligroin, C_6H_6, cold $CHCl_3$. Gradually decomp.
Ethylamide: C_3H_7NS. MW, 89. B.p. 125°/14 mm.
Diethylamide: $C_5H_{11}NS$. MW, 117. Oil. Cryst. on cooling. M.p. below 0°. B.p. 116–17°/14 mm. Spar. misc. with hot H_2O.
Anilide: thioformanilide. Needles from H_2O, plates from EtOH.Aq. M.p. 137·5° decomp.

o-*Toluidide*: needles from EtOH. M.p. 100–1°.

p-*Toluidide*: cryst. from EtOH. M.p. 173·5°.

Willstätter, Wirth, *Ber.*, 1909, **42**, 1911.

Thioglucose.

See Glucothiose.

Thioglycol.

See Ethylene Thioglycol.

Thioglycollic Acid (*Mercaptoacetic acid*)

$HS{\cdot}CH_2{\cdot}COOH$

$C_2H_4O_2S$ MW, 92

F.p. — 16·5°. B.p. 123°/29 mm., 107–8°/16 mm. D^{20} 1·3253. Oxidises in air. NH_3 + $FeCl_3$ ⟶ dark red ⟶ violet col.

Et ester: $C_4H_8O_2S$. MW, 120. B.p. 156–8°, 55°/17 mm. D^{15} 1·0964. *Ag salt*: yellow needles from Me_2CO. M.p. 75–7°. *Hg salt*: needles from Et_2O. M.p. 56·5°. *Bi salt*: yellow cryst. from EtOH. M.p. 82·9°.

Amide: C_2H_5ONS. MW, 91. Needles. M.p. 52°. Very sol. H_2O. Spar. sol. EtOH. Oxidises in air.

Anilide: needles from H_2O or EtOH. M.p. 110·5–111°.

o-*Toluidide*: needles from EtOH.Aq. M.p. 84–5°.

m-*Toluidide*: needles from EtOH.Aq. M.p. 152–3°.

p-*Toluidide*: needles from EtOH. M.p. 125–6°.

S-*Et*: $C_4H_8O_2S$. MW, 120. Oil. F.p. — 8·7°. B.p. 117–18°/11 mm. D^{20}_{20} 1·1518. *Et ester*: $C_6H_{12}O_2S$. MW, 148. B.p. 187–9°. D^4 1·0469. *Amide*: C_4H_9ONS. MW, 119. M.p. 44°.

S-*Propyl*: $C_5H_{10}O_2S$. MW, 134. Oil. *Amide*: m.p. 53°.

S-*Butyl*: $C_6H_{12}O_2S$. MW, 148. Yellow oil. B.p. 140–4°/10–15 mm. Insol. H_2O.

S-*Phenyl*: $C_8H_8O_2S$. MW, 168. Cryst. M.p. 60°. *Amide*: m.p. 103–4°.

S-*Benzyl*: $C_9H_{10}O_2S$. MW, 182. Cryst. M.p. 61–3°.

S-*Acetyl*: yellow oil. B.p. 158–9°/17 mm. *Chloride*: b.p. 93–5°/20 mm.

Holmberg, *J. prakt. Chem.*, 1934, **141**, 93.
Larsson, *Chem. Zentr.*, 1928, II, 234.

Thioguaiacol.

See under Thiocatechol.

Thiohydantoic Acid (*Thioureidoacetic acid, thiocarbaminylglycine*, N-*carboxymethylthiourea*)

$\overset{4}{H_2N}{\cdot}\overset{3}{CS}{\cdot}\overset{2}{NH}{\cdot}\overset{1}{CH_2}{\cdot}COOH$

$C_3H_6O_2N_2S$ MW, 134

Needles or prisms from EtOH. M.p. 170–1° decomp. Very sol. hot H_2O, EtOH. HgO ⟶ hydantoic acid.

Et ester: $C_5H_{10}O_2N_2S$. MW, 162. Cryst. from H_2O. M.p. about 65°. Very sol. H_2O, EtOH, C_6H_6. Insol. Et_2O, pet. ether. 4-N-*Acetyl*: prisms. M.p. 104–5°. 4-N-*Benzoyl*: needles from EtOH. M.p. 128–9°.

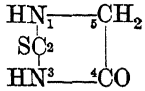
4-N-*Acetyl*: needles. M.p. 205° decomp.

4-N-*Benzoyl*: needles from EtOH, plates from H_2O. M.p. 202°.

4-N-p-*Nitrophenyl*: m.p. 158°.

Wheeler, Nicolet, Johnson, *Am. Chem. J.*, 1911, **46**, 456.
Komatsu, *Chem. Zentr.*, 1911, II, 537.

2-Thiohydantoin (4-*Keto-2-thiotetrahydroiminazole*)



$C_3H_4ON_2S$ MW, 116

White needles from EtOH.Aq. M.p. 228° decomp. Very sol. hot EtOH, Et_2O, alkalis. Spar. sol. H_2O. Gives cryst. K salt. Hot aq. $Ba(OH)_2$ ⟶ thiohydantoic acid.

3-N-*Me*: $C_4H_6ON_2S$. MW, 130. Needles from $CHCl_3$–ligroin. M.p. 161°. Very sol. hot H_2O, EtOH, Et_2O. Spar. sol. $CHCl_3$. Insol. ligroin.

1 : 3-N-*Di-Me*: $C_5H_8ON_2S$. MW, 144. Cryst. from C_6H_6–pet. ether. M.p. 94·5°. Very sol. hot H_2O. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

3-N-*Allyl*: $C_6H_8ON_2S$. MW, 156. Cryst. from ligroin. M.p. 108°. Sol. hot H_2O, EtOH, AcOH. Spar. sol. $CHCl_3$, ligroin, C_6H_6.

3-N-*Phenyl*: $C_9H_8ON_2S$. MW, 192. Yellow plates from EtOH. M.p. 240–2° decomp.

1 : 3-N-*Diphenyl*: $C_{15}H_{12}ON_2S$. MW, 268. Yellow prisms from AcOH, needles from EtOH. M.p. 212°. Sol. hot AcOH. Spar. sol. Et_2O, EtOH, ligroin, C_6H_6.

3-N-*Benzyl*: $C_{10}H_{10}ON_2S$. MW, 206. Cryst. from EtOH. M.p. 128°.

3-N-o-*Tolyl*: yellow plates from EtOH. M.p. 149–50°.

3-N-p-*Tolyl*: yellow cryst. from EtOH. M.p. 228°.

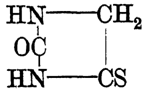
1-N-*Acetyl*: plates from EtOH. M.p. 175–6°.

1-N *Benzoyl*: prisms from EtOH. M.p. 165°.

Picrate: yellow needles from EtOH. M.p. 195–8° decomp.

Johnson, Hill, Bailey, *J. Am. Chem. Soc.*, 1915, **37**, 2406.
Johnson, *Am. Chem. J.*, 1913, **49**, 68.
Marckwald, Neumark, Stelzner, *Ber.*, 1891, **24**, 3285.
Dalgliesh, Mann, *J. Chem. Soc.*, 1947, 559.

4-Thiohydantoin



$C_3H_4ON_2S$ MW, 116

Needles from hot H_2O. Decomp. above 200°. Sol. NaOH ⟶ red sol.

Johnson, Chernoff, *J. Am. Chem. Soc.*, 1912, **34**, 1208.

Thiohydracrylic Acid (2-*Mercaptopropionic acid*)

$HS{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_3H_6O_2S$ MW, 106

Cryst. M.p. 16·8°. B.p. 127–8°, 114–115·5°/13 mm., 85–6°/3 mm. D_4^{20} 1·2199. n_D^{20} 1·4921. Sol. H_2O, EtOH, Et_2O. Ox. ⟶ dithiohydracrylic acid. $FeCl_3$ ⟶ blue col.

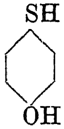
Me ester: b.p. 67–8°/20 mm.

Et ester: b.p. 77·5°/20 mm.

S-*Acetyl*: m.p. 52–4°.

Biilmann, *Ann.*, 1906, **348**, 126.
Lovén, *J. prakt. Chem.*, 1884, **29**, 376.
Holmberg, Schjånberg, *Chem. Abstracts*, 1941, **35**, 2113.
Cheney, Piening, *J. Am. Chem. Soc.*, 1945, **67**, 731.
Gresham, Shaver, U.S.P. 2,474,838, (*Chem. Abstracts*, 1949, **43**, 7506).

Thiohydroquinone (p-*Mercaptophenol*, p-*hydroxythiophenol*)



C_6H_6OS MW, 126

Cryst. with strong odour. M.p. 29–30°. B.p. 166–8°/45 mm., 133–7°/11 mm. Sol. H_2O.

O-*Me ether*: *p*-mercaptoanisole, *p*-methoxythiophenol. C_7H_8OS. MW, 140. B.p. 227°, 89–90°/5 mm. D_4^{25} 1·1313. n_D^{25} 1·5801. Volatile in steam.

S-*Me*: *p*-hydroxythioanisole. Plates from pet. ether. M.p. 84–5°. *Me ether*: *p*-methoxythioanisole. $C_8H_{10}OS$. MW, 154. M.p. 25–6°. B.p. 99–100°/4 mm. D_4^{25} 1·1069. n_D^{25} 1·5764. *Et ether*: *p*-ethoxythioanisole. $C_9H_{12}OS$. MW, 168. M.p. 19–20°. B.p. 98–100°/5 mm. D_4^{25} 1·0693. n_D^{25} 1·5618. *Acetyl*: needles from pet. ether. M.p. 43–4°.

O-*Et ether*: *p*-mercaptophenetole. $C_8H_{10}OS$. MW, 154. B.p. 232–5°. Volatile in steam.

S-*Et*: *p*-hydroxythiophenetole. Cryst. from pet. ether. M.p. 39–41°. *Me ether*: *p*-methoxythiophenetole. $C_9H_{12}OS$. MW, 168. B.p. 103°/5 mm. D_4^{25} 1·0674. n_D^{25} 1·5600. *Et ether*: *p*-ethoxythiophenetole. $C_{10}H_{14}OS$. MW, 182. B.p. 110–12°/6 mm.

S-*Propyl*: propyl *p*-hydroxyphenyl sulphide. $C_9H_{12}OS$. MW, 168. Cryst. from pet. ether. M.p. 33–33·5°. *Me ether*: propyl *p*-methoxyphenyl sulphide. $C_{10}H_{14}OS$. MW, 182. B.p. 110–11°/5 mm. D_4^{25} 1·0424. n_D^{25} 1·5545.

S-*Butyl*: butyl *p*-hydroxyphenyl sulphide. $C_{10}H_{14}OS$. MW, 182. Cryst. from pet. ether. M.p. 49–50°. *Me ether*: butyl *p*-methoxyphenyl sulphide. $C_{11}H_{16}OS$. MW, 196. B.p. 120°/5 mm. D_4^{25} 1·0303. n_D^{25} 1·5445.

S-n-*Amyl*: *n*-amyl *p*-hydroxyphenyl sulphide. $C_{11}H_{16}OS$. MW, 196. Cryst. from pet. ether. M.p. 55–6°. *Me ether*: *n*-amyl *p*-methoxyphenyl sulphide. $C_{12}H_{18}OS$. MW, 210. B.p. 127°/5 mm. D_4^{25} 1·0149. n_D^{25} 1·5380.

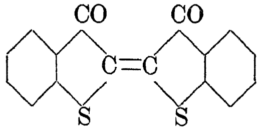
S-n-*Hexyl*: *n*-hexyl *p*-hydroxyphenyl sulphide. $C_{12}H_{18}OS$. MW, 210. Cryst. from pet. ether. M.p. 58–9°. *Me ether*: *n*-hexyl *p*-methoxyphenyl sulphide. $C_{13}H_{20}OS$. MW, 224. B.p. 142°/5 mm. D_4^{25} 0·9975. n_D^{25} 1·5315.

S-*Phenyl*: *p*-hydroxydiphenyl sulphide. $C_{12}H_{10}OS$. MW, 202. Prisms from pet. ether. M.p. 50–1°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Spar. sol. H_2O. *Me ether*: *p*-methoxydiphenyl sulphide. $C_{13}H_{12}OS$. MW, 216. Plates from EtOH.Aq. M.p. 88°. p-*Nitrobenzoyl*: yellow prisms from EtOH. M.p. 74–5°.

S-p-*Tolyl*: *p*-tolyl *p*-hydroxyphenyl sulphide. $C_{13}H_{12}OS$. MW, 216. Plates from pet. ether. M.p. 67–8°. Spar. sol. H_2O. *Me ether*: *p*-tolyl *p*-methoxyphenyl sulphide. $C_{14}H_{14}OS$. MW, 230. Plates from EtOH. M.p. 45–6°. B.p. 181–2°/4 mm.

Miller, Read, *J. Am. Chem. Soc.*, 1933, **55**, 1224.
Suter, Hansen, *J. Am. Chem. Soc.*, 1932, **54**, 4101.
Zincke, Ebel, *Ber.*, 1914, **47**, 1104.
Gattermann, *Ber.*, 1899, **32**, 1148.
Leuckart, *J. prakt. Chem.*, 1890, **41**, 192.

Thioindigo



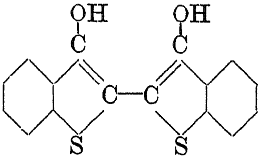
$C_{16}H_8O_2S_2$ MW, 296

Brownish-red needles with bronze reflex from xylene, red cryst. from C_6H_6. Does not melt below 280°. Very sol. hot $PhNO_2$. Spar. sol. hot EtOH, $CHCl_3$, CS_2. Sublimes. Red sol. in xylene shows reddish-yellow fluorescence. Sol. H_2SO_4 ⟶ bluish-green or blue col. Fairly stable to ox. agents. HNO_3 ⟶ thioindigo-S-oxide. Red. ⟶ Thioindigo White.

S-*Oxide*: $C_{16}H_8O_3S_2$. MW, 312. Scarlet cryst. from $CHCl_3$ or xylene. M.p. about 325° decomp. Sol. $CHCl_3$, $PhNO_2$, Py. Spar. sol. EtOH, Me_2CO, C_6H_6. H_2SO_4 ⟶ violet sol.

Jezierski, *Chem. Abstracts*, 1935, **29**, 2161.
Kalle, D.R.P., 194,254, (*Chem. Zentr.*, 1908, I, 1116).
Friedländer, *Ann.*, 1907, **351**, 411.

Thioindigo White (*Leucothioindigo*)



$C_{16}H_{10}O_2S_2$ MW, 298

White ppt. Turns red in air. Sol. most org. solvents. Very sol. dil. NaOH ⟶ sol. which reddens in air. Insol. H_2O.

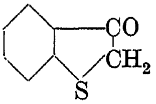
Diacetyl: colourless needles from Ac_2O or xylene. M.p. 248° decomp. (240°).

Monobenzoyl: needles from xylene. M.p. 225° decomp.

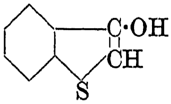
Dibenzoyl: cryst. from xylene. M.p. 227°.

Posner, Wallis, *Ber.*, 1924, **57**, 1673.
Tschilikin, *Chem. Zentr.*, 1916, I, 942.
Béchamp, *Compt. rend.*, 1909, **148**, 1678.

Thioindoxyl (3-*Hydroxythionaphthene*)



or



C_8H_6OS MW, 150

Needles from H_2O. M.p. 71°. Very sol. most org. solvents. Spar. sol. cold H_2O. Sol. alkalis in which it is readily oxidised. Turns red in air.

Me ether: C_9H_8OS. MW, 164. Oil. B.p. 260–1°. *Picrate*: brownish-red needles from EtOH. M.p. 112°.

Et ether: $C_{10}H_{10}OS$. MW, 178. Oil. B.p. 154°/19 mm. $D_4^{17\cdot6}$ 1·1591.

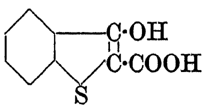
Acetyl: yellow oil. B.p. 165°/18 mm.

Semicarbazone: needles from EtOH. M.p. 224–5°.

p-*Nitrophenylhydrazone*: exists in two forms. (i) Reddish-brown powder. M.p. 185–90°. (ii) Reddish-brown powder. M.p. 251–61°.

McClelland, D'Silva, *J. Chem. Soc.*, 1931, 2974.
Auwers, Thies, *Ber.*, 1920, **53**, 2291.
Friedländer, *Ann.*, 1907, **351**, 408.
Auwers, *Ann.*, 1912, **393**, 379.

Thioindoxylic Acid (3-*Hydroxythionaphthene-2-carboxylic acid, thioindoxyl-2-carboxylic acid*)



$C_9H_6O_3S$ MW, 194

Free acid is very unstable and loses CO_2 readily. Very sol. EtOH, AcOH. Sol. H_2O.

Me ether: $C_{10}H_8O_3S$. MW, 208. Prisms from MeOH. M.p. 173°. Sol. MeOH, EtOH. Spar. sol. C_6H_6, ligroin.

Et ether: $C_{11}H_{10}O_3S$. MW, 222. Prisms from MeOH. M.p. 158°. Very sol. EtOH. Sol. C_6H_6. Spar. sol. ligroin.

Me ester: $C_{10}H_8O_3S$. MW, 208. Cryst. from EtOH.Aq. M.p. 107–8° (104°).

Et ester: $C_{11}H_{10}O_3S$. MW, 222. Cryst. from ligroin. M.p. 73–4°. *Acetyl*: m.p. 105°.

Methylamide: red needles from MeOH. M.p. 122–3°.

Ethylamide: needles from EtOH.Aq. M.p. 135°.

Benzylamide: needles from EtOH. M.p. 134°.

Anilide: needles from EtOH. M.p. 231°. *Acetyl*: needles from EtOH. M.p. 180°.

1 : 1-*Dioxide*: m.p. 137–8°.

Auwers, *Ann.*, 1912, **393**, 372.
Friedländer, *Ann.*, 1907, **351**, 405.

Thioisoamyl Alcohol.
See Isoamyl Mercaptan.

Thioisobutyl Alcohol.
See Isobutyl Mercaptan.

Thioisopropyl Alcohol.
See Isopropyl Mercaptan.

Thiolactic Acid (1-*Mercaptopropionic acid*)

$CH_3{\cdot}CH(SH){\cdot}COOH$

$C_3H_6O_2S$ MW, 106

d-.

B.p. 95–100°/16 mm. $[\alpha]_D^{20}$ + 38·32°.

l-.

Oil. $D^{19\cdot2}$ 1·193. $[\alpha]_D^{15}$ − 45·47°.

dl-.

Oil. B.p. 98·5–99°/14 mm. n_D^{16} 1·4823. Sol. H_2O, EtOH, Et_2O. $FeCl_3$ ⟶ indigo-blue col.

Et ester: $C_5H_{10}O_2S$. MW, 134. Liq. Spar. sol. H_2O. S-*Acetyl*: b.p. 55°/3 mm. n_D^{19} 1·4635.

S-*Benzyl*: prisms. M.p. 73–4°.

Lovén, *J. prakt. Chem.*, 1884, **29**, 368; 1908, **78**, 65.
Biilmann, *Ann.*, 1906, **348**, 124.
Levene, Mikeska, *J. Biol. Chem.*, 1924, **60**, 1.
Larsson, *Chem. Abstracts*, 1928, **22**, 4470.
Fraser, Owen, Shaw, *Biochem. J.*, 1947, **41**, 328.

Thiomalic Acid (*Mercaptosuccinic acid*)

$HS{\cdot}CH{\cdot}COOH$ α
$CH_2{\cdot}COOH$ β

$C_4H_6O_4S$ MW, 150

d-.

M.p. 152–3° (138°). $[\alpha]_D^{17}$ + 64·4° in EtOH.

β-*Monoamide*: $C_4H_7O_3NS$. MW, 149. M.p. 125°. $[\alpha]_D^{18}$ + 82·5° in Me_2CO.

l-.

M.p. 152–3° $[\alpha]_D^{17}$ − 64·8° in EtOH.

β-*Monoamide*: m.p. 125°. $[\alpha]_D^{18}$ − 82·9° in Me_2CO.

dl-.

Cryst. M.p. 151°. Sol. H_2O, EtOH, Me_2CO. Mod. sol. Et_2O. Very spar. sol. C_6H_6. $k = 5{\cdot}23 \times 10^{-4}$ at 25°. $FeCl_3$ ⟶ blue col.

Di-Et ester: $C_8H_{14}O_4S$. MW, 206. Oil. B.p. about 246° part. decomp.

β-*Monoamide*: cryst. from EtOH. M.p. 103°.

S-*Benzyl*: needles from EtOH.Aq. M.p. 181°.

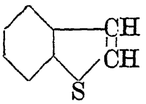
S-*Acetyl*: m.p. 125–6°. *Anhydride*: m.p. 71–3°.

Holmberg, Lenander, *Chem. Zentr.*, 1918, I, 1146.
Biilmann, *Ann.*, 1905, **339**, 371.
Levene, Mikeska, *J. Biol. Chem.*, 1924, **60**, 685.
Holmberg, *Chem. Zentr.*, 1916, I, 968.
Fraser, Owen, Shaw, *Biochem. J.*, 1947, **41**, 328.
Holmberg, Schjånberg, *Chem. Abstracts*, 1941, **35**, 2113.

Thiomorpholine.

See 1 : 4-Thiazan.

Thionaphthene (2 : 3-*Benzthiophene, thiocoumarone*)



C_8H_6S MW, 134

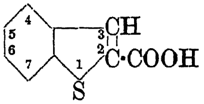
Present in lignite tar. Leaflets with odour resembling naphthalene. M.p. 32°. B.p. 221–2°, 103–5°/20 mm. Sol. common org. solvents. Volatile in steam. Sol. conc. H_2SO_4 with cherry-red col. disappearing on heating. $H_2O_2 \longrightarrow$ S-oxide.

Picrate: yellow needles from EtOH. M.p. 149°.

S-*Oxide*: $C_8H_6O_2S$. MW, 166. M.p. 142°.

Gattermann, Lockhart, *Ber.*, 1893, **26**, 2808.
Bezdrik, Friedländer, Koeniger, *Ber.*, 1908, **41**, 231, 236.
Chmelewsky, Friedländer, *Ber.*, 1913, **46**, 1907.
Weissgerber, Kruber, *Ber.*, 1920, **53**, 1551.
Hansch, Lindwall, *J. Org. Chem.*, 1945, **10**, 381.
Moore, Greensfelder, *J. Am. Chem. Soc.*, 1947, **69**, 2008.

Thionaphthene-2-carboxylic Acid



$C_9H_6O_2S$ MW, 178

Needles from H_2O. M.p. 236° (114°). Very sol. Et_2O. Spar. sol. C_6H_6, cold H_2O. Hot alkalis $\longrightarrow$ thionaphthene.

Me ester: $C_{10}H_8O_2S$. MW, 192. Prisms from EtOH. M.p. 72–3°. B.p. 176–80°/13 mm.

Et ester: $C_{11}H_{10}O_2S$. MW, 206. Cryst. from EtOH. M.p. 36–7°.

Chloride: C_9H_5OClS. MW, 196·5. Leaflets from ligroin. M.p. 88–9°. B.p. 173–5°/19 mm.

Amide: C_9H_7ONS. MW, 177. Needles from H_2O. M.p. 177°.

Hydrazide: leaflets from EtOH. M.p. 184–5°.

Azide: needles from EtOH. M.p. 108° decomp.

Ges. für Teerverwertung, D.R.P., 341,837, (*Chem. Zentr.*, 1921, IV, 1225).
Weissgerber, Kruber, *Ber.*, 1920, **53**, 1561.
Friedländer, Lenk, *Ber.*, 1912, **45**, 2087.

Thionaphthene-3-carboxylic Acid.

Cryst. from EtOH.Aq. M.p. 174–5°. Sol. common org. solvents. Prac. insol. H_2O. Volatile in steam.

Me ester: oil. B.p. 285–7°/750 mm., 165–6°/17 mm.

Et ester: oil. B.p. 304–6°/750 mm., 172–3°/17 mm.

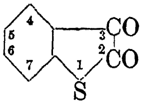
Chloride: m.p. about 50°. B.p. 296–8°/758 mm.

Amide: needles from EtOH. M.p. 197–8°.

Anilide: needles from C_6H_6. M.p. 172–3°.

Komppa, Weekman, *J. prakt. Chem.*, 1933, **138**, 116.
Komppa, *J. prakt. Chem.*, 1929, **122**, 331.

Thionaphthenequinone (2 : 3-*Diketodihydrothionaphthene*, 2 : 3-*diketothiocoumaran, thiocoumarandione*)



$C_8H_4O_2S$ MW, 164

Yellow prisms from EtOH. M.p. 121°. B.p. about 247°. Sol. EtOH, Me_2CO, AcOH, C_6H_6. Spar. sol. cold ligroin. Mod. volatile in steam. Sol. dil. NaOH with orange-yellow col. $\longrightarrow$ 2-mercaptobenzoylformic acid.

2-*Oxime*: yellowish-red cryst. from EtOH. M.p. 170–1°. O-*Me ether*: reddish-yellow needles from EtOH. M.p. 125°. *Acetyl*: yellow needles from xylene. M.p. 174° (168°). *Benzoyl*: yellow plates from C_6H_6. M.p. 170°. *Benzenesulphonyl*: yellow needles from xylene. M.p. 231°. *Phenylhydrazone*: yellow needles from EtOH. M.p. 172°.

3-*Oxime*: yellow needles from EtOH.Aq. M.p. 186°.

3-*Phenylhydrazone*: 3-benzeneazo-2-hydroxythionaphthene. Orange-red cryst. from EtOH. M.p. 165–6°.

Di-phenylhydrazone: orange-red needles from C_6H_6. M.p. 199–200°.

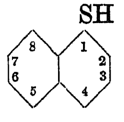
3-p-*Nitrophenylhydrazone*: red cryst. from xylene. M.p. 271–2°.

2-*Anil*: yellowish-red needles from EtOH. M.p. 151–2°.

2-p-*Tolil*: brownish-red needles from EtOH. M.p. 159°.

Kalle, D.R.P., 241,623, (*Chem. Zentr.*, 1912, I, 174).
Bezdrik, Friedländer, Koeniger, *Ber.*, 1908, **41**, 235.

1-Thionaphthol (1-*Naphthyl mercaptan*, 1-*mercaptonaphthalene*, α-*thionaphthol*)



$C_{10}H_8S$ MW, 160

B.p. 208·5°/200 mm., 187·2°/50 mm., 161°/20 mm., 144·8°/10·3 mm. D_4^{23} 1·1549. Sol. EtOH, Et_2O. Spar. sol. aq. alkalis. Volatile in steam.

Me ether: *see* Methyl 1-naphthyl sulphide.

Et ether: ethyl 1-naphthyl sulphide. $C_{12}H_{12}S$. MW, 188. B.p. 175–6°/25 mm., 167–167·5°/15 mm. D_4^0 1·1198, D_4^{50} 1·0797.

Phenyl ether: *see* Phenyl 1-naphthyl sulphide.

o-*Tolyl ether*: *o*-tolyl 1-naphthyl sulphide. $C_{17}H_{14}S$. MW, 250. Oil. B.p. 227·5°/11 mm. D_4^{15} 1·1504.

m-*Tolyl ether*: *m*-tolyl 1-naphthyl sulphide. Oil. B.p. 229°/11 mm. D_4^{15} 1·1445.

p-*Tolyl ether*: *p*-tolyl 1-naphthyl sulphide. Cryst. from EtOH. M.p. 40·5°. B.p. 232·5°/11 mm.

Naphthyl ether: *see* Dinaphthyl sulphide.

Benzyl ether: benzyl 1-naphthyl sulphide. Leaflets from EtOH. M.p. 78–80°.

Acetyl: pale yellow liq. B.p. 200–3°/25 mm., 188°/15 mm. D_4^{50} 1·1519.

Bourgeois, *Rec. trav. chim.*, 1899, **18**, 441; *Ber.*, 1895, **28**, 2328.
Fichter, Tamm, *Ber.*, 1910, **43**, 3033.
Leuckart, *J. prakt. Chem.*, 1890, **41**, 216.
Lazier, Signaigo, Wise, U.S.P. 2,402,645, (*Chem. Abstracts*, 1946, **40**, 5761).
Signaigo, U.S.P. 2,402,686, (*Chem. Abstracts*, 1946, **40**, 5766).

2-Thionaphthol (2-*Naphthyl mercaptan*, 2-*mercaptonaphthalene*, β-*thionaphthol*).

Cryst. from EtOH. M.p. 81°. B.p. 286°, 210·5°/100 mm., 189°/50 mm., 162·7°/20 mm., 153·5°/15 mm., 146·3°/10·3 mm. Very sol. EtOH, Et_2O, pet. ether. Spar. sol. H_2O. Spar. volatile in steam.

Me ether: *see* Methyl 2-naphthyl sulphide.

Et ether: ethyl 2-naphthyl sulphide. M.p. 16°. B.p. 170·5°/15 mm.

Phenyl ether: *see* Phenyl 2-naphthyl sulphide.

o-*Tolyl ether*: *o*-tolyl 2-naphthyl sulphide. Oil. B.p. 229·5°/11 mm. D_4^{15} 1·1420.

m-*Tolyl ether*: *m*-tolyl 2-naphthyl sulphide. Needles from EtOH.Aq. M.p. 60°. B.p. 235°/11 mm.

p-*Tolyl ether*: *p*-tolyl 2-naphthyl sulphide. Leaflets from EtOH.Aq. M.p. 68°. B.p. 237°/11 mm.

Naphthyl ether: *see* Dinaphthyl sulphide.

Acetyl: m.p. 53·5°. B.p. 191°/15 mm.

Zincke, Eismayer, *Ber.*, 1918, **51**, 755.
Leuckart, *J. prakt. Chem.*, 1890, **41**, 219.
Fichter, Tamm, *Ber.*, 1910, **43**, 3034.
Lazier, Signaigo, U.S.P. 2,402,641, (*Chem. Abstracts*, 1946, **40**, 5768).

Thioneine.
See Ergothioneine.

Thionessal.
See Tetraphenylthiophene.

Thionylaniline

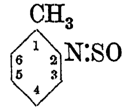
$C_6H_5N{:}SO$

C_6H_5ONS MW, 139

Yellow liq. with acrid aromatic odour. B.p. 200°. D^{15} 1·2360. Sol. EtOH. Decomp. by H_2O, dil. acids, alkalis, ⟶ aniline + SO_2. Cl in pet. ether ⟶ 2 : 4 : 6-trichloroaniline hydrochloride.

Michaelis, *Ber.*, 1891, **24**, 746.

Thionyl-*o*-toluidine



C_7H_7ONS MW, 153

Liq. B.p. 184°/100 mm.

Michaelis, *Ann.*, 1893, **274**, 226.

Thionyl-*m*-toluidine.

Yellow oil. B.p. 220°. Volatile in steam.

Michaelis, *Ann.*, 1893, **274**, 226.

Thionyl-*p*-toluidine.

Yellow cryst. M.p. 9°. B.p. 224°. D^{15} 1·1685.

Michaelis, *Ann.*, 1893, **274**, 226.

Thio-oxalic Acid

HO·CS·COOH or HOOC·CO·SH

$C_2H_2O_3S$ MW, 106

Free acid does not exist.

Di-Me ester: $C_4H_6O_3S$. MW, 134. B.p. 50–3°/21 mm.

Di-Et ester: $C_6H_{10}O_3S$. MW, 162. B.p. 217°. D^0 1·1446. Hyd. ⟶ oxalic acid.

S-p-*Tolyl ester*: $C_9H_8O_3S$. MW, 196. Plates or needles from pet. ether. M.p. 100° decomp. Very sol. Et_2O, EtOH. Spar. sol. pet. ether. Sol. H_2O with decomp. *Anilide*: needles from EtOH. M.p. 137°.

Amide: *see* Thio-oxamic Acid.

Diamide: *see under* Thio-oxamic Acid.

Nitrile: C_2HONS. MW, 87. *Anilide*: orange-yellow needles. M.p. 82°. o-*Toluidide*: m.p. 64°.

Anilide: yellow needles from H_2O, yellow plates from EtOH.Aq. M.p. 101–2°. *Amide*: oxanilic acid thioamide. Yellow prisms from EtOH. M.p. 176°.

Dianilide: thio-oxanilide. Yellow needles from AcOH or EtOH. M.p. 144–5°.

Di-o-*toluidide*: yellow needles and plates from EtOH. M.p. 126°.

Di-m-*toluidide*: yellow needles and plates from EtOH. M.p. 88–9°.

Di-p-*toluidide*: yellow needles from EtOH. M.p. 153–4°.

Reissert, *Ber.*, 1904, **37**, 3720.
Morley, Saint, *J. Chem. Soc.*, 1883, **43**, 400.

Thio-oxamic Acid

$H_2N{\cdot}CS{\cdot}COOH$

$C_2H_3O_2NS$ MW, 105

Exists only as salts and esters.

Me ester: $C_3H_5O_2NS$. MW, 119. Yellow needles. M.p. 86°. Very sol. EtOH, Et_2O. Sol. H_2O.

Et ester: $C_4H_7O_2NS$. MW, 133. Yellow prisms. M.p. 63°. Very sol. hot H_2O, EtOH, Et_2O.

Isobutyl ester: $C_6H_{11}O_2NS$. MW, 161. Yellow needles or prisms from EtOH. M.p. 58°. Sol. EtOH, Et_2O. Spar. sol. H_2O.

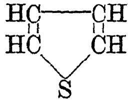
Amide: thio-oxamide. $C_2H_4ON_2S$. MW, 104. Yellow needles from EtOH. Spar. sol. cold H_2O, cold EtOH.

Nitrile: cyanthioformamide. $C_2H_2N_2S$. MW, 86. Yellow needles. M.p. 87–90° decomp. Sol. H_2O. Very unstable.

Anilide: thio-oxanilic acid amide. Yellow needles from EtOH. M.p. 169–70°.

Weddige, *J. prakt. Chem.*, 1874, **10**, 200; **9**, 133.

Thiophene



C_4H_4S MW, 84

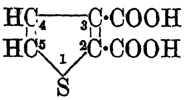
F.p. − 37·1°. M.p. 29·8°. B.p. 84°. D_4^{20} 1·0617. n_D^{20} 1·5246. Heat of comb. C_p 670·9 Cal., C_v 669·5 Cal., (vapour) C_p 610·6 Cal. Red hot tube ⟶ 2 : 2′-dithienyl + 3 : 3′-dithienyl. Sol. H_2SO_4 ⟶ red ⟶ deep brown col. H_2SO_4 + nitrite ⟶ blue col. Isatin + H_2SO_4 ⟶ indophenin. Thalline in pet. ether + HNO_3 ⟶ intense violet col. ⟶ red ⟶ yellow: disappears with H_2O.

Jurjew, *Ber.*, 1936, **69**, 440, 1002.
Phillips, *Organic Syntheses*, 1932, XII, 72.
I.G., B.P. 305,603.
Arnold, B.P. 535,583, (*Chem. Abstracts*, 1942, **36**, 1338).
Cooper, U.S.P. 2,415,627, (*Chem. Abstracts*, 1947, **41**, 2754).
Barger, Easson, *J. Chem. Soc.*, 1938, 2100.

Thiophene-acetic Acid.
See Thienylacetic Acid.

Thiophene-carboxylic Acid.
See Thiophenic Acid.

Thiophene-2 : 3-dicarboxylic Acid



$C_6H_4O_4S$ MW, 172

Prisms. M.p. 270°.

Di-Me ester: $C_8H_8O_4S$. MW, 200. M.p. 58°.

Diamide: needles. M.p. 228°.

Anhydride: m.p. 140°.

Dinitrile: needles from ligroin. M.p. 140°.

Imide: cryst. by sublimation. M.p. 204°.

Linstead, Noble, Wright, *J. Chem. Soc.*, 1937, 911.

Thiophene-2 : 4-dicarboxylic Acid (*Thiophene-α : β′-dicarboxylic acid*).

Decomp. at 280° with part. sublimation. Very sol. hot H_2O, spar. sol. cold.

Di-Me ester: plates from EtOH.Aq. M.p. 120–1°.

Di-Et ester: $C_{10}H_{12}O_4S$. MW, 228. M.p. 35–6°.

Zelinsky, *Ber.*, 1887, **20**, 2021.
Linstead, Noble, Wright, *J. Chem. Soc.*, 1937. 911.

Thiophene-2 : 5-dicarboxylic Acid (*Thiophene-α : α′-dicarboxylic acid*).

Cryst. powder. Does not melt below 350°. Sol. Et_2O. Spar. sol. H_2O. Sublimes at 150–300°.

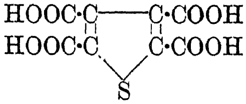
Di-Me ester: needles from Et_2O, prisms from EtOH. M.p. 151° (145°).

Di-Et ester: needles from EtOH. M.p. 51·5°. Sol. EtOH.

Dinitrile: $C_6H_2N_2S$. MW, 134. Cryst. from Et_2O. M.p. 92–92·5°.

Hinsberg, *Ber.*, 1912, **45**, 2414.
Jaekel, *Ber.*, 1886, **19**, 190.
Campaigne, Foye, *J. Am. Chem. Soc.*, 1948, **70**, 3941.

Thiophene-tetracarboxylic Acid



$C_8H_4O_8S$ MW, 260

Tetra-Me ester: $C_{12}H_{12}O_8S$. MW, 316. Prisms from EtOH. M.p. 126–8°. Very sol. hot EtOH, AcOEt. Spar. sol. hot H_2O.

Michael, *Ber.*, 1895, **28**, 1635.

Thiophenetidine.
See under p-Aminothiophenol.

Thiophenetole (*Ethyl phenyl sulphide*)

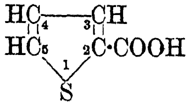
$C_6H_5{\cdot}S{\cdot}CH_2{\cdot}CH_3$

$C_8H_{10}S$ MW, 138

Liq. with unpleasant odour. B.p. 204°, 69°/6 mm. D_4^{15} 1·024. $n_D^{22\cdot5}$ 1·5662. $KMnO_4$ ⟶ ethyl phenyl sulphone.

Otto, *Ber.*, 1880, **13**, 1275.
Vogel, *J. Chem. Soc.*, 1948, 1820.

α-Thiophenic Acid (*Thiophene-2-carboxylic acid*)



$C_5H_4O_2S$ MW, 128

Needles from H_2O. M.p. 129–30°. Very sol. hot H_2O, EtOH, Et_2O. Sol. $CHCl_3$. Spar. sol. pet. ether. Sol. 140 parts H_2O at 25°. Has irritating odour. $k = 3{\cdot}2 \times 10^{-4}$ at 25°. Heat of comb. C_v 645·4 Cal., C_p 646·3 Cal. Red. ⟶ tetrahydrothiophene-2-carboxylic acid. Dist. Ca salt ⟶ 2 : 2′-dithienyl ketone. Isatin + H_2SO_4 ⟶ blue sol.

Et ester: $C_7H_8O_2S$. MW, 156. B.p. 218°, 115°/25 mm., 96°/18 mm. $D_4^{16\cdot3}$ 1·1623.

Chloride: C_5H_3OClS. MW, 146·5. B.p. 190°.

Amide: C_5H_7ONS. MW, 127. Cryst. powder from EtOH. M.p. 180° (174°). Spar. sol. Et_2O.

Anhydride: $C_{10}H_6O_3S_2$. MW, 238. Cryst. M.p. 62°. B.p. 218–20°/15 mm.

Nitrile: 2-cyanthiophene. C_5H_3NS. MW, 109. Oil. B.p. 192°. Volatile in steam.

Hydrazide: needles from H_2O. M.p. 136°.

Azide: yellow plates. M.p. 37°.

Anilide: plates. M.p. 140°.

Steinkopf, Ohse, *Ann.*, 1924, **437**, 18.
Voerman, *Rec. trav. chim.*, 1907, **26**, 296.
Nahnsen, *Ber.*, 1884, **17**, 2195.
Campaigne, Foye, *J. Am. Chem. Soc.*, 1948, **70**, 3941.
Challenger, Gibson, *J. Chem. Soc.*, 1940, 305.
Gilman, Shirley, *J. Am. Chem. Soc.*, 1949, **71**, 1870.

β-Thiophenic Acid (*Thiophene-3-carboxylic acid*).

Needles from H_2O. M.p. 138·4°. Sol. 230 parts H_2O at 25°. Sublimes. Volatile in steam. $k = 7{\cdot}8 \times 10^{-5}$ at 25°. Gives indophenin reaction.

Et ester: b.p. 207–8°/736 mm. D_4^{27} 1·1799. n_D^{20} 1·5230.

p-*Bromophenacyl ester*: m.p. 129–30°.

Chloride: m.p. 51–2°. B.p. 203–4°/748 mm., 110–11°/36 mm.

Amide: needles from Et_2O. M.p. 179–80°. Spar. sol. Et_2O.

Voerman, *Rec. trav. chim.*, 1907, **26**, 297.
Rinkes, *Rec. trav. chim.*, 1936, **55**, 991.
Campaigne, LeSuer, *J. Am. Chem. Soc.*, 1948, **70**, 1555.
Steinkopf, Schmitt, *Ann.*, 1938, **533**, 264.
Weinmayr, U.S.P. 2,462,697, (*Chem. Abstracts*, 1949, **43**, 3850).

Thiophenine.
See 2-Aminothiophene.

Thiophenol (*Phenyl mercaptan, mercaptobenzene*)

$C_6H_5{\cdot}SH$

C_6H_6S MW, 110

Liq. with penetrating odour. B.p. 169·5°, 103·6°/100 mm., 86·2°/50 mm., 77°/30 mm. D_4^{25} 1·0728, D_4^{50} 1·0491, D_4^{75} 1·0254. $n_D^{23\cdot2}$ 1·5861. Forms metallic salts. Oxidises in air, especially in alcoholic ammonia solution, to diphenyl disulphide.

Me ether: *see* Thioanisole.

Et ether: *see* Thiophenetole.

Phenyl ether: *see* Diphenyl sulphide.

Acetyl: *see* S-Phenyl ester under Thioacetic Acid.

Benzoyl: *see* S-Phenyl ester under Thiobenzoic Acid.

Adams, Marvel, *Organic Syntheses*, Collective Vol. I, 490.
Winter, *Am. Chem. J.*, 1904, **31**, 572.
Signaigo, U.S.P. 2,402,686, (*Chem. Abstracts*, 1946, **40**, 5766).

Thiophos 3422.
See Parathion.

Thiophosgene.
See Thiocarbonyl chloride.

Thiopropionic Acid

$CH_3{\cdot}CH_2{\cdot}CO{\cdot}SH$ or $CH_3{\cdot}CH_2{\cdot}CS{\cdot}OH$

C_3H_6OS MW, 90

Liq. with pungent sulphur-like odour.

S-*Me ester*: C_4H_8OS. MW, 104. B.p. 119–20°.

O-*Et ester*: $C_5H_{10}OS$. MW, 118. B.p. 130–2°. D_4^{19} 0·9451. n_D^{19} 1·46281. Insol. H_2O.

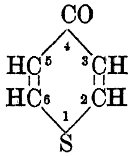
Amide: C_3H_7NS. MW, 89. Leaflets from C_6H_6. M.p. 41–3°. Very sol. C_6H_6. Spar. sol. H_2O, EtOH, Et_2O.

Anilide: needles from AcOH.Aq. M.p. 67–67·5°.

Delépine, *Bull. soc. chim.*, 1911, **9**, 907.
Weigert, *Ber.*, 1903, **36**, 1009.
Hubacher, *Ann.*, 1890, **259**, 229.

Thiopropyl Alcohol.
See Propyl Mercaptan.

Thio-γ-pyrone



C_5H_4OS MW, 112

Prisms from CCl_4. M.p. 110°. Mod. sol. H_2O, EtOH, Et_2O. Spar. sol. ligroin. Sol. conc. H_2SO_4.

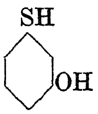
HCl salt: m.p. 135° (not sharp).

Add. comp. with $HgCl_2$: needles from H_2O. M.p. 189°.

Dioxide: m.p. 173–4°.

Arndt, Bekir, *Ber.*, 1930, **63**, 2395.

Thioresorcinol (m-*Hydroxythiophenol*, m-*mercaptophenol*)



C_6H_6OS MW, 126

Cryst. with penetrating odour. M.p. 16–17°. B.p. 168°/35 mm. Sol. common org. solvents except pet. ether. Spar. sol. H_2O. Volatile in steam.

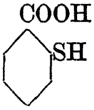
O-*Me ether*: *m*-mercaptoanisole, *m*-methoxythiophenol. C_7H_8OS. MW, 140. Liq. B.p. 224–5°, 112–14°/20 mm., 96–100°/9–10 mm.

S-*Me ether*: *m*-hydroxythioanisole, methyl *m*-hydroxyphenyl sulphide. Cryst. M.p. 15°. B.p. 224° slight decomp., 148–51°/14 mm. Sol. common org. solvents except pet. ether. Volatile in steam.

S-*Et ether*: *m*-hydroxythiophenetole, ethyl *m*-hydroxyphenyl sulphide. $C_8H_{10}OS$. MW, 154. B.p. 238–9°, 104–5°/9–10 mm.

Watson, Dutt, *J. Chem. Soc.*, 1922, **121**, 2415.
Zincke, Ebel, *Ber.*, 1914, **47**, 927.
Szathmáry, *Ber.*, 1910, **43**, 2487.
Mauthner, *Ber.*, 1906, **39**, 3596.

Thiosalicylic Acid (o-*Mercaptobenzoic acid*)



$C_7H_6O_2S$ MW, 154

Leaflets or needles from EtOH or AcOH. M.p. 164–5°. Sol. EtOH, AcOH. Spar. sol. hot H_2O. Oxidises in air ⟶ diphenyl disulphide 2 : 2′-dicarboxylic acid. $FeCl_3$ in alc. sol. ⟶ blue col. Alk. $KMnO_4$ ⟶ 2-sulphobenzoic acid.

Me ether: *see* S-Methylthiosalicylic Acid.

Et ether: S-ethylthiosalicylic acid. $C_9H_{10}O_2S$. MW, 182. Yellowish cryst. M.p. 134–5°. Sol. EtOH, AcOH. Very spar. sol. H_2O. *Et ester*: $C_{11}H_{14}O_2S$. MW, 210. Cryst. M.p. 27–8°. B.p. 152–3°/10 mm.

Phenyl ether: *see* Diphenyl sulphide 2-carboxylic Acid.

Nitrophenyl ether: *see* Nitrodiphenyl sulphide 2-carboxylic Acid.

p-*Tolyl ether*: cryst. from MeOH. M.p. 215–16°.

2-*Naphthyl ether*: leaflets from EtOH. M.p. 200–1°.

Benzyl ether: needles. M.p. 189°.

Acetyl: thioaspirin. Needles from C_6H_6. M.p. 125°.

Me ester: $C_8H_8O_2S$. MW, 168. Oil. B.p. 262–3°/728 mm. Volatile in steam.

Phenyl ester: thiosalol. $C_{13}H_{10}O_2S$. MW, 230. Cryst. from MeOH. M.p. 91°. Sol. EtOH, Et_2O. Spar. sol. ligroin.

Allen, Mackay, *Organic Syntheses*, 1932, XII, 76.
Mayer, *Ber.*, 1909, **42**, 1134.
Geigy, Swiss P. 227,349, (*Chem. Abstracts*, 1949, **43**, 3039).

Thiosalol.
See under Thiosalicylic Acid.

Thiosemicarbazide

$H_2N{\cdot}CS{\cdot}NH{\cdot}NH_2$

CH_5N_3S MW, 91

Needles from H_2O. M.p. 181–3°.

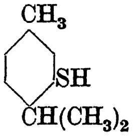
Hydrochloride: m.p. 186–90°.

Derivatives are given elsewhere under their own names.

Freund, Schander, *Ber.*, 1896, **29**, 2501.
Bambas, U.S.P. 2,450,406, (*Chem. Abstracts*, 1949, **43**, 1057).

Thiosinamine.
See Allylthiourea.

Thiothymol (3-*Mercapto*-p-*cymene*, 3-*methyl*-6-*isopropylthiophenol, thymyl mercaptan*)



$C_{10}H_{14}S$ MW, 166

B.p. 230–1°.

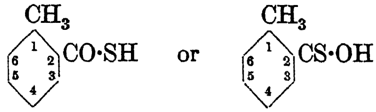
$Hg(C_{10}H_{13}S)_2$: greenish cryst. from EtOH. M.p. 78°.

Fittica, *Ann.*, 1874, **172**, 305, 325.

Thiotolene.
See Methylthiophene.

Thio-α-toluic Acid.
See Phenylthioacetic Acid.

Thio-o-toluic Acid (o-*Methylthiobenzoic acid*)



C_8H_8OS MW, 152

Yellow oil. B.p. 133°/35 mm. D_{25}^{25} 1·1451. Sol. EtOH, Et_2O. Insol. H_2O.

S-*Et ester*: $C_{10}H_{12}OS$. MW, 180. Yellow oil. B.p. 133°/15 mm. D_{25}^{25} 1·0513. Sol. EtOH, Et_2O. Insol. H_2O.

Amide: *o*-methylthiobenzamide. C_8H_9NS. MW, 151. M.p. 88°.

Phenylhydrazide: m.p. 116–18°.

Sachs, Reid, *J. Am. Chem. Soc.*, 1916, **38**, 2748.

Thio-*p*-toluic Acid (p-*Methylthiobenzoic acid*).

Greenish prisms from pet. ether. M.p. 43·5–44°. B.p. 131°/15 mm. Very volatile in steam.

S-*Et ester*: b.p. 150°/18 mm. D_{25}^{25} 1·0708. Sol. EtOH, Et_2O. Insol. H_2O.

O-*Et ester*: b.p. 140–5°/70 mm. D_4^{20} 0·9992.

S-p-*Nitrobenzyl ester*: yellow cryst. from EtOH. M.p. 97°.

Amide: *p*-methylthiobenzamide. Cryst. M.p. 168°. N-*Benzoyl*: red prisms from EtOH. M.p. 135–6°.

Anilide: yellow needles from EtOH. M.p. 140–1°.

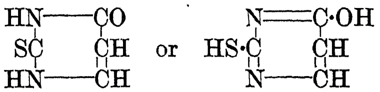
p-*Toluidide*: yellow cryst. from Me_2CO. M.p. 165–6°.

Sachs, Reid, *J. Am. Chem. Soc.*, 1916, **38**, 2748.

Thiotoluidine.

See Diaminoditolyl sulphide.

2-Thiouracil



$C_4H_4ON_2S$ MW, 128

Prisms from H_2O or EtOH. M.p. about 340° decomp. Sol. NH_3, alkalis. Spar. sol. H_2O, EtOH.

Wheeler, Bristol, *Am. Chem. J.*, 1905, **33**, 458.

Wheeler, Liddle, *Am. Chem. J.*, 1908, **40**, 550.

Thiourea (*Thiocarbamide, thiocarbonic acid diamide*)

$$H_2N{\cdot}CS{\cdot}NH_2 \rightleftharpoons HN{:}C(SH){\cdot}NH_2$$

Isothiourea

CH_4N_2S MW, 76

Rhombohedra or needles from EtOH. M.p. 180°. Sol. H_2O, EtOH. Spar. sol. Et_2O. Reacts neutral. Forms add. comps. with metallic salts and oxides. Prolonged heating at 170° $\longrightarrow$ NH_4CNS. $Ag_2O \longrightarrow$ dicyandiamide.

Hydrochloride: m.p. 136–7°.

B_4,NH_4Cl: cryst. from EtOH. M.p. 154°.

B_4,NH_4Br: needles from EtOH. M.p. 180°.

B_4,NH_4I: cryst. from EtOH. M.p. 186°.

S-*Me*: *see* S-Methylisothiourea.

N : S-*Diacetyl*: yellow prisms from AcOH.Aq. M.p. 153°.

Tri-Me: prisms from C_6H_6–ligroin. M.p. 87–8°. Sol. H_2O, EtOH, $CHCl_3$.

N-*Tetra-Me*: m.p. 78°. B.p. 245°.

N-*Tetra-Et*: b.p. 264–6°, 130°/12 mm. D_4^{18} 0·9662. n_D^{26} 1·5225.

Triphenyl: needles from EtOH. M.p. 152°. Spar. sol. cold EtOH.

N-*Tetraphenyl*: needles from EtOH. M.p. 194·5–195·5°. Sol. C_6H_6. Mod. sol. Et_2O. Spar. sol. EtOH. Insol. H_2O.

Other thiourea derivatives are given separately elsewhere.

Kirchoff, Akonjans, *Chem. Zentr.*, 1935, I, 2165.

Giua, *Chem. Abstracts*, 1925, **19**, 1561.

I.G., F.P., 655,457, (*Chem. Zentr.*, 1929, II, 487).

Ciba, U.S.P., 2,006,762, (*Chem. Abstracts*, 1935, **29**, 5463).

Werner, *J. Chem. Soc.*, 1912, **101**, 2185.

Schenck, *Z. physiol. Chem.*, 1912, **77**, 370.

Delépine, *Bull soc. chim.*, 1902, **27**, 814.

I.G., D.R.P., 526,799, (*Chem. Abstracts*, 1931, **25**, 4892).

Gebhardt, *Ber.*, 1884, **17**, 2092.

Cooper, U.S.P. 2,353,997, (*Chem. Abstracts*, 1944, **38**, 5846).

Arundale, Mikeska, U.S.P. 2,337,882, (*Chem. Abstracts*, 1944, **38**, 3296).

Thiourethane

$$H_2N{\cdot}CS{\cdot}OC_2H_5 \text{ or } H_2N{\cdot}CO{\cdot}SC_2H_5$$

C_3H_7ONS MW, 105

O-*Ester*: Xanthogenamide.

Pyramids. M.p. 40–1° (16°). Decomp. on dist. D_4^{20} 1·069. n_D^{20} 1·520.

N-*Di-Me*: $C_5H_{11}ONS$. MW, 133. M.p. 15°. D_4^{20} 1·028. n_D^{20} 1·5075.

N-*Di-Et*: $C_7H_{15}ONS$. MW, 161. B.p. 114°/20 mm.

N-*Acetyl*: prisms from H_2O. M.p. 101°.

N-*Isovaleryl*: prisms from Me_2CO. M.p. 56°.

N-*Carbomethoxyl*: needles. M.p. 83°.

S-*Ester*:

Plates. M.p. 102° (109°). Sol. EtOH, Et_2O, hot H_2O. Insol. cold H_2O.

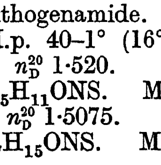
N-*Acetyl*: prisms from C_6H_6. M.p. 98°.

Battegay, Hégazi, *Helv. Chim. Acta*, 1933, **16**, 1005.

Homberg, *Chem. Abstracts*, 1930, **24**, 2111.

Wheeler, Barnes, *Am. Chem. J.*, 1899, **22**, 148.

Thioxan (1 : 4-*Oxthian*)



C_4H_8OS MW, 104

M.p. − 17°. B.p. 147°, 109·8°/233 mm., 86·5°/97 mm., 69·9°/47 mm. D_4^{20} 1·1178. n_D^{20} 1·5081. Spar. sol. H_2O.

Ethiodide: yellow cryst. from EtOH. M.p. 85°.

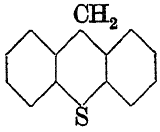
Dibromide: m.p. 75–80° decomp.

Di-iodide: cryst. from Et_2O. M.p. 66–7°.

Mercurichloride: needles from EtOH. M.p. 171°.

Johnson, *J. Chem. Soc.*, 1933, 1530.
Fromm, Ungar, *Ber.*, 1923, **56**, 2288.
Clarke, *J. Chem. Soc.*, 1912, **101**, 1806.
Yur'ev, Novitskiĭ, *Chem. Abstracts*, 1949, **43**, 2624.

Thioxanthene (*Diphenylmethane sulphide, dibenzthiopyran*)



$C_{13}H_{10}S$ MW, 198

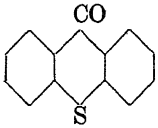
Cryst. from EtOH–$CHCl_3$. M.p. 128°. B.p. 340°/730 mm. Sol. $CHCl_3$. Spar. sol. Et_2O, cold EtOH. Sublimes. $CrO_3 \longrightarrow$ benzophenone sulphone.

S-*Oxide*: diphenylmethane sulphoxide. Plates from pet. ether. M.p. 109–10°.

S-*Dioxide*: *see* Diphenylmethane sulphone.

Graebe, Schultefs, *Ann.*, 1891, **263**, 12.

Thioxanthone (9-*Ketothioxanthene*)



$C_{13}H_8OS$ MW, 212

Yellow needles from $CHCl_3$. M.p. 209° (207°). Sublimes. Sol. AcOH, $CHCl_3$, CS_2, C_6H_6. Insol. H_2O. Red. $\longrightarrow$ thioxanthene. $CrO_3 \longrightarrow$ benzophenone sulphone.

S-*Dioxide*: *see* Benzophenone sulphone.

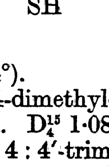
Hydrazone: yellow leaflets from EtOH–C_6H_6. M.p. 115°.

Gomberg, Britton, *J. Am. Chem. Soc.*, 1921, **43**, 1945.
Graebe, Schultefs, *Ann.*, 1891, **263**, 8.

Thioxene.

See Dimethylthiophene.

***m*-4-Thioxylenol** (4-*Mercapto*-m-*xylene*, 2 : 4-*dimethylphenyl mercaptan*)



$C_8H_{10}S$ MW, 138

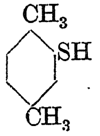
B.p. 207–8° (214°).

S-*Phenyl*: 2 : 4-dimethyldiphenyl sulphide. B.p. 172·5°/11 mm. D_4^{15} 1·0817.

S-p-*Tolyl*: 2 : 4 : 4′-trimethyldiphenyl sulphide. B.p. 188°/11 mm. D_4^{15} 1·0614.

Gattermann, *Ber.*, 1899, **32**, 1147.

***p*-2-Thioxylenol** (2-*Mercapto*-p-*xylene*, 2 : 5-*dimethylphenyl mercaptan*)



$C_8H_{10}S$ MW, 138

B.p. 211–12° (205°).

S-*Phenyl*: 2 : 5-dimethyldiphenyl sulphide. B.p. 171°/11 mm. D_4^{15} 1·0795.

S-p-*Tolyl*: 2 : 5 : 4′-trimethyldiphenyl sulphide. B.p. 185°/11 mm. D_4^{15} 1·0606.

S-*Benzyl*: m.p. 35°. B.p. 200°/15 mm.

Gattermann, *Ber.*, 1899, **32**, 1147.

Thiuram disulphide

$H_2N{\cdot}CS{\cdot}S{\cdot}S{\cdot}CS{\cdot}NH_2$

$C_2H_4N_2S_4$ MW, 184

Plates from Me_2CO–$CHCl_3$. M.p. 153° decomp. Sol. Me_2CO, hot EtOH. Insol. H_2O, Et_2O, $CHCl_3$. Heat $\longrightarrow CS_2 + NH_4CNS$.

Freund, Bachrach, *Ann.*, 1895, **285**, 201.

Threitol (1 : 2 : 3 : 4-*Tetrahydroxybutane*)

```
          H    OH
HO·CH2-C——C-CH2OH
         OH    H
```

$C_4H_{10}O_4$ MW, 122

d-.

Needles from EtOH. M.p. 88·5–89°. $[\alpha]_D$ + 4·3° in H_2O, − 11·1° in 95% EtOH. Very sol. boiling EtOH.

Di-benzylidene deriv.: m.p. 231°.

Orsellinate: *see* Montagnetol.

l-.

Needles from EtOH. M.p. 88°. $[\alpha]_D$ − 4·4° in H_2O, + 11° in 95% EtOH. Very sol. H_2O, boiling EtOH.

Di-benzylidene deriv.: m.p. 231°.

dl-.

Cryst. from EtOH. M.p. 72°. Very sol. H_2O.

Anhydride: m.p. 40°. B.p. 59–60°/30 mm.

Tetra-acetyl: m.p. 53°.

Di-benzylidene deriv.: m.p. 220°.

Orsellinate: *see* Montagnetol.

Pariselle, *Ann. chim. phys.*, 1911, **24**, 401.
Maquenne, Bertrand, *Compt. rend.*, 1901, **132**, 1566.
Bertrand, *Ann. chim. phys.*, 1904, **3**, 207.
Maquenne, *Ann. chim. phys.*, 1901, **24**, 406.
Ruff, *Ber.*, 1901, **34**, 1371.
Lucas, Baumgarten, *J. Am. Chem. Soc.*, 1941, **63**, 1653.

Threonic Acid (*Trihydroxybutyric acid*)

```
         OH   H
HO·CH2-C——C-COOH
          H   OH
```

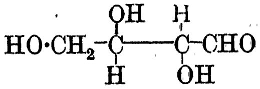
$C_4H_8O_5$ MW, 136

d-.
Syrup. $[\alpha]_D$ about $-30°$ in H_2O.
Phenylhydrazide: laminæ from EtOH. M.p. 157–8°. $[\alpha]_D^{20}$ $-29°$ to $-31°$.
Brucine salt: plates. M.p. 214°. $[\alpha]_D^{20}$ $-32{\cdot}4°$ in H_2O.
Quinine salt: needles from EtOH. M.p. 168°. $[\alpha]_D^{20}$ $-116{\cdot}99°$ in H_2O.
Strychnine salt: plates from dil. EtOH. M.p. 115–21°. $[\alpha]_D^{20}$ $-28{\cdot}5°$ in H_2O.
l-.
Syrup. $[\alpha]_D^{19}$ $+9{\cdot}54°$.
Phenylhydrazide: m.p. 157°. $[\alpha]_D^{20}$ about $+29°$.
Me ester: $C_5H_{10}O_5$. MW, 150. *Tri-Me ether*: b.p. 120°/13 mm. n_D^{15} 1·4275. $[\alpha]_{5780}^{18}$ $+49°$ in MeOH, $+31°$ in H_2O. D^{15} 1·090.
Amide: $C_4H_9O_4N$. MW, 135. M.p. 105·5–107°. $[\alpha]_D^{20}$ $+58°$ in H_2O. *Tri-Me ether*: m.p. 78°. $[\alpha]_{5780}^{20}$ $+44°$ in H_2O, $+68°$ in MeOH.
Brucine salt: m.p. 208–9°. $[\alpha]_D^{20}$ $-28{\cdot}1°$ in H_2O.
Quinine salt: m.p. 169·5–170·5°. $[\alpha]_D^{22}$ $-116{\cdot}7°$ in H_2O.
Strychnine salt: m.p. 182–4°. $[\alpha]_D^{21}$ $-18{\cdot}5°$ in H_2O.
Lactone: m.p. 65–8°. $[\alpha]_D^{21}$ $+30{\cdot}7°$ in H_2O.
dl-.
M.p. 98–9°.
Amide: m.p. 116°.
Phenylhydrazide: m.p. 167·5°.

Jensen, Upson, *J. Am. Chem. Soc.*, 1925, **47**, 3023.
Nef, *Ann.*, 1914, **403**, 265.
Nef, Hedenburg, Glattfeld, *J. Am. Chem. Soc.*, 1917, **39**, 1642.
Braun, *J. Am. Chem. Soc.*, 1932, **54**, 1137.
Haworth, Hirst, Smith, *J. Chem. Soc.*, 1934, 1558.
Glattfeld, Hoen, *J. Am. Chem. Soc.*, 1935, **57**, 1407.
Reichstein, Grüssner, Bosshard, *Helv. Chim. Acta*, 1935, **18**, 605.
Herbert *et al.*, *J. Chem. Soc.*, 1933, 1284.
Glattfield, Rietz, *J. Am. Chem. Soc.*, 1940, **62**, 974.
Lucas, Baumgarten, *J. Am. Chem. Soc.*, 1941, **63**, 1653.

Threonine.
See 2-Hydroxy-1-aminobutyric Acid.

Threose (*Trihydroxybutyraldehyde*)

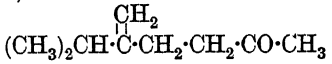


$C_4H_8O_4$ MW, 120

d-.
M.p. 126–32°. Very hygroscopic. $[\alpha]_D^{22}$ $+29{\cdot}09° \longrightarrow 19{\cdot}59°$ in H_2O. NaHg ⟶ *d*-erythritol.
Phenylosazone: m.p. 164–5°.
Phenylbenzylhydrazone: needles from C_6H_6. M.p. 194·5°.
Acetone deriv.: m.p. 84°. $[\alpha]_D^{22}$ $-15{\cdot}27°$ in Me_2CO.
Diacetamide deriv.: prisms. M.p. 166°. $[\alpha]_D$ $-10{\cdot}9°$ in H_2O.
Diacetyl: m.p. 140–2°. $[\alpha]_D^{23}$ $+83{\cdot}52° \longrightarrow 34{\cdot}31°$ in $CHCl_3$. Reduces Fehling's.
Triacetyl: m.p. 118°. $[\alpha]_D^{20}$ $+35{\cdot}5°$ in $CHCl_3$.
l-.
Obtained only in solution. $[\alpha]_D^{2}$ $-24{\cdot}6°$ in H_2O. Reduces Fehling's in the cold.
Phenylosazone: cryst. from C_6H_6. M.p. 165–6°.
Diacetamide deriv.: cryst. from 95% EtOH. M.p. 165–6°.

Maquenne, *Ann. chim. phys.*, 1901, **24**, 404.
Ruff, *Ber.*, 1901, **34**, 1370.
Deulofeu, *J. Chem. Soc.*, 1929, 2458; *J. Am. Chem. Soc.*, 1936, **58**, 855.
Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 1016.
Mendive, *Chem. Abstracts*, 1932, **26**, 2433.
Freudenberg, *Ber.*, 1932, **65**, 168.
Hockett, *J. Am. Chem. Soc.*, 1934, **56**, 994.
Lake, Glattfield, *J. Am. Chem. Soc.*, 1944, **66**, 1091.

Thujaketone (*Tanacet-ketone*, 2-*isopropyl*-1-*hexenone*-5)

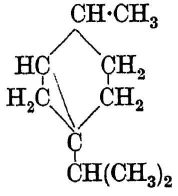


$C_9H_{16}O$ MW, 140

B.p. 184–6°. D^{20} 0·854. n_D 1·441.
Oxime: b.p. 118–20°/15 mm.
Semicarbazone: m.p. 143°.

Tiemann, Semmler, *Ber.*, 1897, **30**, 439.
Wallach, *Ann.*, 1893, **272**, 116.
Birch, Earl, *Chem. Abstracts*, 1939, **33**, 3361.

Thujane (*Sabinane*)

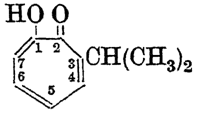


$C_{10}H_{18}$ MW, 138

d-.
Mobile oil with faint odour. B.p. 157°/758 mm. D_4^{20} 0·8139. n_D^{20} 1·43759. $[\alpha]_D$ $+62{\cdot}03°$.

Tschugaev, Fomin, *Compt. rend.*, 1910, **151**, 1058.
Henderson, Robertson, *J. Chem. Soc.*, 1923, **123**, 1715.
Zelinsky, Turowa-Pollak, *Ber.*, 1929, **62**, 2868.
Guha, Nath, *Ber.*, 1937, **70**, 931.
Guha, Krishnamurthy, *ibid.*, 2112.

α-Thujaplicin (*3-Isopropyltropolone*)



$C_{10}H_{12}O_2$ MW, 164

Occurs in heartwood of *Thuja plicata*, D. Don. Needles from pet. ether. M.p. 82°. Antifungal.

Gripenberg, *Acta Chem. Scand.*, 1948, 2, 639.

β-Thujaplicin (*Hinokitiol, 4-isopropyltropolone*).

Occurs in oil of wood of Hinoki tree (*Chamaecyparis obtusa*, Lieb et Zucc.) and in heartwood of *Thuja plicata*. Cryst. from pet. ether. M.p. 52–52·5°. $FeCl_3$ ⟶ hinokitin. Antifungal.

Cr complex: m.p. 230°.
Cu complex: m.p. 177°.
Ni complex: m.p. 242°.

Nozoe, *Bull. Chem. Soc. Japan*, 1936, **11**, 295.
Anderson, Gripenberg, *Acta Chem. Scand.*, 1948, 2, 644.
Nozoe, *Nature*, 1951, **167**, 1055.

γ-Thujaplicin (*5-Isopropyltropolone*).

Occurs in heartwood of *Thuja plicata*, D. Don. Needles from pet. ether. M.p. 82°. Antifungal.

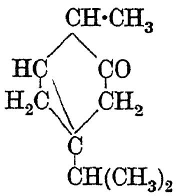
Cu complex: m.p. 259–60°.

Erdtman, Gripenberg, *Acta Chem. Scand.*, 1948, **2**, 625.

Thujol.

See Thujyl Alcohol.

Thujone



$C_{10}H_{16}O$ MW, 152

Two stereoisomers are known.

α-. *l*-Thujone.

Constituent of many essential oils. Oil with fresh odour. B.p. 199–201°, 103–4°/40 mm., 74·5°/9 mm. D^{20} 0·9152. n_D^{25} 1·4490. $[\alpha]_D^{18}$ − 19·94° (homogeneous). Heat ⟶ carvacrol + carvotanacetone. Boiling alc. H_2SO_4 ⟶ β-thujone ⟶ isothujone.

Oxime: liq. $[\alpha]_D^{19}$ − 29·25° in Et_2O.

Semicarbazone: (i) Prisms from MeOH. M.p. 186–8°. $[\alpha]_D$ + 64·4° in EtOH. (ii) Amorphous. M.p. 100–10°. $[\alpha]_D^{20}$ + 53·71°.

2 : 4-*Dinitrophenylhydrazone*: orange-yellow plates from EtOH. M.p. 116–17°. $[\alpha]_D^{16}$ + 44° in $CHCl_3$.

m-*Nitrobenzoylhydrazone*: m.p. 156–156·3°.

β-. Tanacetone, *d*-isothujone.

Constituent of many essential oils. Oil with odour resembling menthol. B.p. 201–2°, 76°/10 mm. $D_4^{15\cdot1}$ 0·9193. $n_D^{13\cdot6}$ 1·4540. $[\alpha]_D^{20}$ + 77·33°. Heat of comb. C_v 1429·9 Cal. Alc. KOH ⟶ α-thujone. Heat ⟶ carvotanacetone. Boiling dil. H_2SO_4 ⟶ isothujone. Na + EtOH ⟶ thujyl alcohol. Forms cryst. bisulphite comp.

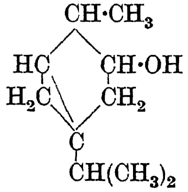
Oxime: tanacetone oxime. Prisms. M.p. 54–5°. B.p. 135–6°/20 mm. $[\alpha]_D^{11}$ + 105·1° in MeOH. *Benzoyl*: cryst. from MeOH. M.p. 52–3°.

Hydrazone: b.p. 149°/35 mm. D_0^{22} 0·9504. n_D^{22} 1·4952. $[\alpha]_D$ + 123·75°.

Semicarbazone: (i) Hexagonal cryst. M.p. 174–5°. $[\alpha]_D^{15}$ + 218·04° in MeOH. (ii) Rhombic cryst. M.p. 170–2°.

Challenger, *Industrial Chemist*, 1928, **4**, 315.
Paolini, *Chem. Abstracts*, 1926, **20**, 1072.
Östling, Roth, *Ber.*, 1913, **46**, 313.
Rose, Livingstone, *J. Am. Chem. Soc.*, 1912, **34**, 201.
Guha, Nath, *Ber.*, 1937, **70**, 931.
Guha, Muthanna, *Ber.*, 1938, **71**, 2671.
Short, Read, *J. Chem. Soc.*, 1938, 2016; 1939, 1040.

Thujyl Alcohol (*β-Thujyl alcohol, tanacetyl alcohol, thujol*)



$C_{10}H_{18}O$ MW, 154

Eight active and four racemic isomers are possible. Occurs free and combined as ester in several essential oils. Characteristic constants for the commercial product (mixture of isomers) are:—b.p. 208–10° (220°), 92·5°/13 mm. D^{25} 0·9266. n_D^{25} 1·4621. Heat of comb. C_v 1477·1 Cal.

Me ether: $C_{11}H_{20}O$. MW, 168. D_4^{20} 0·8771. n_D 1·44541.

Two dextro- and two lævorotatory modifications are described in the literature.

d-. *d*-Isothujyl alcohol.

Sweet-smelling, viscous liq. B.p. 206°, 103°/16 mm. D_4^{20} 0·9187. n_D^{16} 1·4625. $[\alpha]_D^{14}$ +106·70°. CrO_3 ⟶ β-thujone.

Hydrogen phthalate: needles from pet. ether. M.p. 120°. $[\alpha]_D$ + 91·3° in EtOH. *Ag salt*: m.p. 85–6°. *Strychnine salt*: needles from EtOH.Aq. M.p. 177–8°. $[\alpha]_D$ + 36·8°.

3 : 5-*Dinitrobenzoate*: long colourless needles. M.p. 92°. $[\alpha]_D^{17}$ + 96·75° in $CHCl_3$.

d-. δ-Thujyl alcohol. (There is doubt about the stereochemical purity).

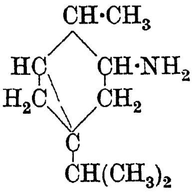
B.p. 206°. n_D 1·4759. $[\alpha]_D$ +50·01°.
Hydrogen phthalate: cryst. from pet. ether. M.p. 95–6°. $[\alpha]_D$ + 2·3° in EtOH.

l-. *l*-Thujyl alcohol.
Long fine needles. M.p. 66–7°. $[\alpha]_D^{16}$ − 20·5° in MeOH. $[\alpha]_D^{17}$ − 22·5° in EtOH.
p-*Nitrobenzoyl*: glistening plates. M.p. 101°. $[\alpha]_D^{15}$ − 32·25° in $CHCl_3$.
3 : 5-*Dinitrobenzoyl*: long fine needles. M.p. 106°. $[\alpha]_D^{19}$ − 24·5° in $CHCl_3$.

l-. *l*-Neothujyl alcohol.
M.p. 22–3°. B.p. 96°/13 mm. D_4^{25} 0·9223. n_D^{25} 1·4624. $[\alpha]_D$ −8·8° in EtOH.
p-*Nitrobenzoyl*: m.p. 90°. $[\alpha]_D$ −12·5° in $CHCl_3$.
3 : 5-*Dinitrobenzoyl*: m.p. 103°. $[\alpha]_D$ −10° in $CHCl_3$.

Short, Read, *J. Chem. Soc.*, 1938, 2016; 1939, 1040.

Thujylamine



$C_{10}H_{19}N$ MW, 153

As with thujyl alcohol, eight active forms and four racemic mixtures are possible. Four thujylamines are described.

A.
B.p. 199·6°. $[\alpha]_D^{25}$ + 27·80°.
B,H₂SO₄: hexagonal plates. M.p. 242° decomp. $[\alpha]_D^{25}$ + 42·77° in H_2O.
B,HNO₃: hemihydrate, massive plates. M.p. 105°. $[\alpha]_D^{25}$ + 35·97° in H_2O.
B,H₂C₂O₄: monohydrate, long needles. Decomp. on heating. $[\alpha]_D^{25}$ + 36·10° in H_2O.
Benzoyl: prisms. M.p. 73–5°. $[\alpha]_D^{25}$ + 91·44° in MeOH.
p-*Toluenesulphonyl*: thick needles. M.p. 194·7°. $[\alpha]_D^{25}$ + 27·91°.

B. (*d*-Isothujylamine ?).
B.p. 193·4°. $[\alpha]_D^{25}$ + 94·94°.
B,H₂SO₄: cubic cryst. of monohydrate. M.p. 153° decomp. $[\alpha]_D^{25}$ + 55·25° in H_2O.
B,HNO₃: prisms. M.p. 176·9°. $[\alpha]_D^{25}$ + 70·48° in H_2O.
B,H₂C₂O₄: long needles. M.p. 167°. $[\alpha]_D^{25}$ + 62·50° in H_2O.
Benzoyl: needles. M.p. 131·5°. $[\alpha]_D^{25}$ + 87·74° in MeOH.
p-*Toluenesulphonyl*: long needles. M.p. 170–1°. $[\alpha]_D^{25}$ + 41·60°.

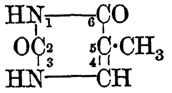
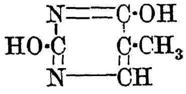
C.
B.p. 196·7°. $[\alpha]_D^{25}$ − 14·15°.
B,H₂SO₄: plates of tetrahydrate. M.p. 243° decomp. $[\alpha]_D^{25}$ + 3·47° in H_2O.
B,HNO₃: flat needles. M.p. 150°. $[\alpha]_D^{25}$ + 2·60° in H_2O.
B,H₂C₂O₄: plates. M.p. 200–1°.
Benzoyl: plates. M.p. 94·5°. $[\alpha]_D^{25}$ − 12·16° in MeOH.

D. (*l*-Thujylamine ?).
B.p. 202·2°. $[\alpha]_D^{25}$ − 22·07°.
B,H₂SO₄: scaly plates of monohydrate. M.p. 263° decomp. $[\alpha]_D^{25}$ − 16·66° in H_2O.
B,HNO₃: long needles. M.p. 159–60°. $[\alpha]_D^{25}$ − 15·18° in H_2O.
B₂,H₂C₂O₄: plates. M.p. 235° decomp. $[\alpha]_D^{25}$ − 12·37° in H_2O.
p-*Nitrobenzoyl*: needles. M.p. 146·5°. $[\alpha]_D^{25}$ − 51·25°.
p-*Toluenesulphonyl*: long needles. M.p. 198·6°. $[\alpha]_D^{25}$ − 10·40°.

Dickison, Ingersoll, *J. Am. Chem. Soc.*, 1939, **61**, 2477.

Thymacetin.
See under 6-Aminothymol.

Thymine (5-*Methyluracil*)


or


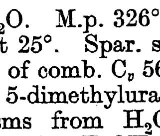
$C_5H_6O_2N_2$ MW, 126

Plates from H_2O. M.p. 326° (318–20°). Sol. 250 parts H_2O at 25°. Spar. sol. EtOH, Et_2O. Sublimes. Heat of comb. C_v 566·4 Cal.
1-N-*Me*: 1 : 5-dimethyluracil. $C_6H_8O_2N_2$. MW, 140. Prisms from H_2O. M.p. 202–5° decomp. Very sol. hot EtOH, Me_2CO.
3-N-*Me*: 3 : 5-dimethyluracil. Needles or prisms from H_2O. M.p. 280–2°.
1 : 3-N-*Di-Me*: 1 : 3 : 5-trimethyluracil. $C_7H_{10}O_2N_2$. MW, 154. Needles from EtOH. M.p. 153°. Very sol. H_2O, $CHCl_3$. Spar. sol. Et_2O, pet. ether.

Bergmann, Currie, *J. Am. Chem. Soc.*, 1933, **55**, 1734.
Johnson, Clapp, *J. Biol. Chem.*, 1908, **5**, 56.
Scherp, *J. Am. Chem. Soc.*, 1946, **68**, 912.

Thyminose.
See 2-Ribodesose.

Thymohydroquinone (2 : 5-*Dihydroxy*-p-*cymene, thymoquinol, hydrothymoquinone*)



$C_{10}H_{14}O_2$ MW, 166

Occurs free and combined as ether in several essential oils. Prisms. M.p. 143° (139·5°). B.p. 290°. Very sol. EtOH, Et_2O. Sol. hot H_2O. Insol. C_6H_6. Sublimes. Heat of comb. C_p 1308·6 Cal., C_v 1308·1 Cal. Ox. ⟶ thymoquinone.

Di-Me ether: $C_{12}H_{18}O_2$. MW, 194. B.p. 248–50°, 118°/12 mm. D^{22} 0·998. n_D^{22} 1·51339.

Diacetyl: m.p. 73–5°.

Dibenzoyl: pale yellow needles from EtOH. M.p. 141–2°.

Phenylurethane: leaflets from EtOH. M.p. 232–3°.

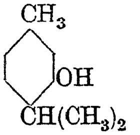
α-*Naphthylurethane*: cryst. from EtOH. M.p. 147–8°.

> Sherk, *Chem. Zentr.*, 1921, III, 218.
> Sabatier, Mailhe, *Compt. rend.*, 1908, **146**, 458.

Thymohydroxycuminic Acid.

See 3-Hydroxycuminic Acid.

Thymol (3-*Hydroxy*-p-*cymene*, 3-*hydroxy*-4-*isopropyltoluene*, 3-*methyl*-6-*isopropylphenol*, 4-*isopropyl*-m-*cresol*)



$C_{10}H_{14}O$ MW, 150

Constituent of numerous essential oils. Plates with odour of thyme from AcOEt, AcOH or Me_2CO. M.p. 51·5°. B.p. 233·5°. Sol. 1176 parts H_2O at 19·4°. Very sol. EtOH, Et_2O, AcOH, $CHCl_3$, C_6H_6. Heat of comb. C_p 1353·75 Cal. Crit. temp. 425·1°. Red. ⟶ menthol + menthone. Ox. ⟶ thymohydroquinone or dithymol. $KNO_2 + H_2SO_4$ ⟶ green ⟶ blue col. Possesses antiseptic properties.

Me ether: $C_{11}H_{16}O$. MW, 164. Occurs naturally. Liq. with ethereal odour. B.p. 211–12°/745 mm., 94–6°/15 mm. D_4^{14} 0·9388.

Et ether: $C_{12}H_{18}O$. MW, 178. B.p. 224–8°. D_0^0 0·9334.

Propyl ether: $C_{13}H_{20}O$. MW, 192. B.p. 243°. D_0^0 0·9276.

Isopropyl ether: b.p. 226·5–228°. D_4^{15} 0·915. n_D^{51} 1·4988.

Butyl ether: $C_{14}H_{22}O$. MW, 206. B.p. 258·3°. D_0^0 0·9230

d-*Amyl ether*: $C_{15}H_{24}O$. MW, 220. B.p. 250–60°. D^{18} 0·934. n_D^{17} 1·5056. $[\alpha]_D^{18}$ + 4·17°.

Isoamyl ether: b.p. 242–3°/746·5 mm. n_D^{14} 1·4923.

Phenyl ether: $C_{16}H_{18}O$. MW, 226. B.p. 297°/766 mm., 176°/25 mm. D^{15} 1·0113.

Benzyl ether: $C_{17}H_{20}O$. MW, 240. B.p. 221–3°/35 mm. D_{18}^{18} 1·0063. n_D^{20} 1·5511.

p-*Nitrobenzyl ether*: $C_{17}H_{19}O_3N$. MW, 285. Cryst. from EtOH.Aq. M.p. 85·5°.

Formyl: b.p. 81°/2 mm. D^0 1·015. n_D^{20} 1·49606.

Acetyl: b.p. 242–3°. D^0 1·009.

Oxalate: needles from EtOH. M.p. 61°.

Benzoyl: plates. M.p. 33°.

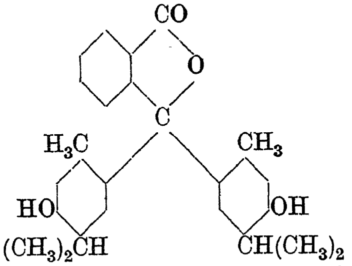
3 : 5-*Dinitrobenzoyl*: needles. M.p. 103·2°.

Phenylurethane: cryst. from EtOH.Aq. M.p. 106·5–107°.

α-*Naphthylurethane*: m.p. 160°.

> Rheinische Kampher-Fabrik., B.P. 308,681 (*Chem. Zentr.*, 1931, II, 1492).
> Austerweil, Lemay, *Bull. soc. chim.*, 1927, **41**, 454.
> Frisch, D.R.P., 615,470, (*Chem. Zentr.*, 1936, **107**, I, 883; *Chem. Abstracts*, 1935, **29**, 6252).
> Hund, U.S.P., 1,967,440.

Thymolphthalein (2′ : 2″-*Dimethyl*-5′ : 5″-*di-isopropylphenolphthalein*)



$C_{28}H_{30}O_4$ MW, 430

Prisms from EtOH. M.p. 246–7°. Indicator.

> Willstätter, Waldschmidt-Leitz, *Ber.*, 1923, **56**, 488.

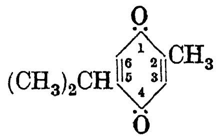
Thymomenthone.

See under Menthone.

Thymoquinol.

See Thymohydroquinone.

Thymoquinone (1-*Methyl*-4-*isopropylcyclohexadiene*-3 : 6-*dione*, 2-*methyl*-5-*isopropyl*-1 : 4-*benzoquinone*)



$C_{10}H_{12}O_2$ MW, 164

Occurs in oil from wood of *Callitris quadrivalvis*, Vent., and *Monarda fistulosa*. Bright yellow tablets with penetrating odour. M.p. 45·5°. B.p. 232°. Very sol. EtOH, Et_2O. Sol. $CHCl_3$, C_6H_6. Spar. sol. H_2O. Volatile in steam. Heat of comb. C_p 1274·6 Cal., C_v 1273·4 Cal. In light ⟶ dithymoquinone. EtOH + light ⟶ thymohydroquinone. Red. ⟶ thymohydroquinone.

Thymohydroquinone add. comp.: dark violet needles. M.p. 64°.

1-*Oxime*: *see* 6-Nitrosothymol.

4-*Oxime*: *see* 5-Nitrosocarvacrol.

1 : 4-*Dioxime*: $C_{10}H_{14}O_2N_2$. MW, 194. Prisms from EtOH. Darkens at 200°. Decomp. at 235°. *Diacetyl*: plates or needles from ligroin. M.p. 110°. *Dibenzoyl*: needles from C_6H_6. M.p. 199–200°.

1-*Semicarbazone*: yellow needles from EtOH. M.p. 201–2° decomp.

1 : 4-*Disemicarbazone*: cryst. from AcOH. M.p. 237°.

1-*Phenylsemicarbazone*: dark yellow needles from AcOH. M.p. 204–5°.

1:4-*Diphenylsemicarbazone*: dark yellow cryst. powder. Decomp. at 242°.

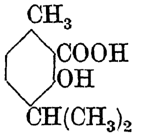
1-*Phenylhydrazone*: reddish-yellow needles from EtOH. M.p. 93°.

1-o-*Nitrophenylhydrazone*: red needles from MeOH. M.p. 145°.

1-[2 : 4-*Dinitrophenyl*]-*hydrazone*: dark red needles from EtOH. M.p. 179–80°.

Tseng, Hu, Chu, *J. Chinese Chem. Soc.*, 1934, **2**, 151.
Kremers, Hixon, Wakeman, *Organic Syntheses*, 1926, VI, 92.
Liebermann, Iljinski, *Ber.*, 1885, **18**, 3194, 3220.
Sumerford, Dalton, *J. Am. Chem. Soc.*, 1944, **66**, 1330.

o-Thymotinic Acid (3-*Hydroxy*-4-*isopropyl*-o-*toluic acid*, 3-*hydroxy*-1-*methyl*-4-*isopropylbenzene*-2-*carboxylic acid*, 6-*methyl*-3-*isopropylsalicylic acid*)



$C_{11}H_{14}O_3$ MW, 194

Needles from H_2O, C_6H_6 or ligroin. M.p. 127°. Sol. 10,000 parts H_2O at 20°. Sol. EtOH, Et_2O, AcOH, $CHCl_3$, C_6H_6. Volatile in steam. $FeCl_3$ ⟶ dark blue col. Strong bactericide.

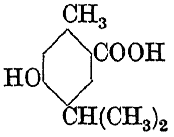
Me ester: $C_{12}H_{16}O_3$. MW, 208. Yellow oil. B.p. 142°/18·5 mm.

Et ester: $C_{13}H_{18}O_3$. MW, 222. Yellow oil. B.p. 153°/18·5 mm.

Acetonyl ester: $C_{14}H_{18}O_4$. MW, 250. Needles from EtOH. M.p. 75°. Very sol. hot EtOH, Et_2O, Me_2CO, C_6H_6. Has local anæsthetic action.

Spallino, Provenzal, *Gazz. chim. ital.*, 1909, **39**, 326.

p-Thymotinic Acid (5-*Hydroxy*-4-*isopropyl*-o-*toluic acid*, 5-*hydroxy*-1-*methyl*-4-*isopropylbenzene*-2-*carboxylic acid*)



$C_{11}H_{14}O_3$ MW, 194

Plates from EtOH.Aq. M.p. 157°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. hot H_2O. No col. with $FeCl_3$.

Me ether: 2-methyl-5-isopropylanisic acid. $C_{12}H_{16}O_3$. MW, 208. Needles from EtOH. M.p. 138–9°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. cold H_2O. *Cu salt*: blue cryst. from EtOH–ligroin. Decomp. at 190°. *Et ester*: $C_{14}H_{20}O_3$. MW, 236. Liq. with unpleasant odour. B.p. 163–4°/13 mm. D_4^{25} 1·032. *Amide*: $C_{12}H_{17}O_2N$. MW, 207. Needles from EtOH. M.p. 158–9°. Sol. EtOH, AcOH. Spar. sol. Et_2O, ligroin. *Nitrile*: $C_{12}H_{15}ON$. MW, 189. Cryst. from ligroin. M.p. 69–70°. B.p. 158–60°/16 mm. Very sol. EtOH, Et_2O, $CHCl_3$. Sol. ligroin.

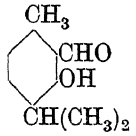
Et ether: $C_{13}H_{18}O_3$. MW, 222. Prisms from EtOH. M.p. 159°. Spar. sol. H_2O. *Amide*: $C_{13}H_{19}O_2N$. MW, 221. Needles from EtOH. M.p. 127°.

Me ester: $C_{12}H_{16}O_3$. MW, 208. Cryst. from Et_2O or C_6H_6. M.p. 97–8°.

Nitrile: $C_{11}H_{13}ON$. MW, 175. Cryst. from C_6H_6. M.p. 115–16°.

Houben, Fischer, *Ber.*, 1931, **64**, 245.
Grignard, Bellet, Courtot, *Ann. chim.*, 1915, **4**, 50.
Gattermann, *Ann.*, 1888, **244**, 69.
Kobek, *Ber.*, 1883, **16**, 2102.

o-Thymotinic Aldehyde (3-*Hydroxy*-4-*isopropyl*-o-*toluic aldehyde*, 2-*aldehydothymol*, 6-*hydroxy*-2-*methyl*-5-*isopropylbenzaldehyde*)



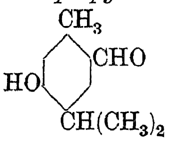
$C_{11}H_{14}O_2$ MW, 178

B.p. 130°/15 mm.

Semicarbazone: m.p. 198°.

Duff, *J. Chem. Soc.*, 1941, 547.

p-Thymotinic Aldehyde (5-*Hydroxy*-4-*isopropyl*-o-*toluic aldehyde*, 6-*aldehydothymol*, 4-*hydroxy*-2-*methyl*-5-*isopropylbenzaldehyde*)



$C_{11}H_{14}O_2$ MW, 178

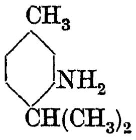
Needles from hot H_2O. M.p. 133°. Very sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. hot H_2O. Forms cryst. bisulphite comp. No col. with $FeCl_3$.

Me ether: 2-methyl-5-isopropylanisaldehyde. $C_{12}H_{16}O_2$. WM, 192. B.p. 278°. Very sol. EtOH, Et_2O, C_6H_6. *Anil*: plates from ligroin. M.p. 80°.

Anil: pale yellow needles. M.p. 142°.

Gattermann, Berchelmann, *Ber.*, 1898, **31**, 1767.
Kobek, *Ber.*, 1883, **16**, 2097.

Thymylamine (5-*Methyl*-2-*isopropylaniline*, 3-*amino*-p-*cymene*)



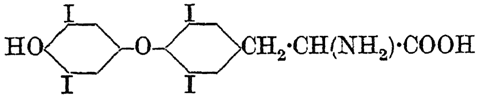
$C_{10}H_{15}N$ MW, 149

Oil with unpleasant odour. B.p. 230°. Volatile in steam. Misc. with EtOH, Et_2O. Spar. sol. H_2O.

Acetyl: needles from EtOH. M.p. 112°.

Lloyd, *Ber.*, 1887, **20**, 1259.
Cooke, Macbeth, *J. Chem. Soc.*, 1937, 1593.

Thyroxine



$C_{15}H_{11}O_4NI_4$ MW, 777

dl-.

Isolated from thyroid gland. Needles by addition of AcOH to alk. EtOH.Aq. sol. Darkens at 220°. M.p. 231–3° (250°) decomp. Very spar. sol. H_2O. Insol. org. solvents. Sol. HN_3, and alc. alkalis. Increases rate of basal metabolism.

Me ester: $C_{16}H_{13}O_4NI_4$. MW, 791. Prisms from EtOH.Aq. M.p. 156°. Spar. sol. H_2O, org. solvents except EtOH. *B,HCl*: needles from EtOH–HCl. M.p. 221·5°. *Me ether*: m.p. 224°. N-*Acetyl*: plates from anisole. M.p. 208–9° decomp. N-*Chloroacetyl*: prisms from C_6H_6. M.p. 159–60°.

Et ester: $C_{17}H_{15}O_4NI_4$. MW, 805. N-*Diacetyl*: needles. M.p. 216–17° (230°).

N-*Acetyl*: cryst. from AcOH.Aq. M.p. 210–15° decomp.

N-*Chloroacetyl*: cryst. from AcOH.Aq. M.p. 201–2° decomp.

l-.

M.p. 235°. $[\alpha]_{5461}$ − 4·45° in EtOH–NaOH.

d-.

$[\alpha]^{21}_{5461}$ + 2·97° in EtOH–NaOH.

Harington, Slater, *Biochem. J.*, 1930, **24**, 456.
Kendall, *Thyroxine*, (Chem. Catalog. Co., New York, 1929).
Ashley, Harington, *Biochem. J.*, 1929, **23**, 1178; 1928, **22**, 1436.
Harington, *Biochem. J.*, 1928, **22**, 1429; 1926, **20**, 293.
Harington, Barger, *Biochem. J.*, 1927, **21**, 169.

Tiglic Acid (cis-1 : 2-*Dimethylacrylic acid*, 1-*methylcrotonic acid*, 1-*ethylidenepropionic acid*)

$CH_3{\cdot}CH$
$\quad\|$
$CH_3{\cdot}C{\cdot}COOH$

$C_5H_8O_2$ MW, 100

Occurs as glyceride in croton oil, as ester in Roman cumin oil. Tablets and columns from H_2O. M.p. 64°. B.p. 198·5°. D_4^{76} 0·9641. n_α^{76} 1·43297. Sol. hot H_2O. Heat of comb. 626·6 Cal. $k = 0{\cdot}957 \times 10^{-5}$ at 25°. HI (+ P) $\longrightarrow$ 1-methylbutyric acid.

Me ester: $C_6H_{10}O_2$. MW, 114. B.p. 139·4–139·6°/766 mm. D_4^{20} 0·94980. n_D^{20} 1·4370.

Et ester: $C_7H_{12}O_2$. MW, 128. B.p. 156°/752 mm., 80·5–81·5°/45 mm., 55·5°/11 mm. $D_4^{19\cdot5}$ 0·9247. $n_{He}^{19\cdot5}$ 1·43554.

Isobutyl ester: b.p. 180°.

Isoamyl ester: $C_{10}H_{18}O_2$. MW, 170. Occurs in Roman cumin oil. B.p. 204–5°.

d-*Citronellyl ester*: $C_{15}H_{26}O_2$. MW, 238. Liq. with pleasant odour. B.p. 144–5°/7 mm. D_{15}^{15} 0·9090.

Geranyl ester: $C_{15}H_{24}O_2$. MW, 236. Liq. with pleasant odour. B.p. 149–51°/7 mm. D_{15}^{15} 0·9279.

p-*Bromophenacyl ester*: cryst. from EtOH. M.p. 67·9°.

Chloride: C_5H_7OCl. MW, 118·5. B.p. 45°/12 mm.

Anilide: cryst. from pet. ether. M.p. 77°.

2-*Naphthylamide*: cryst. M.p. 96°.

Michael, Ross, *J. Am. Chem. Soc.*, 1933, **55**, 3692.
Auwers, Wissebach, *Ber.*, 1923, **56**, 715.
Auwers, *Ann.*, 1923, **432**, 70.
Blaise, *Bull. soc. chim.*, 1903, **29**, 330.
Weizmann, Sulzbacher, Bergmann, *J. Am. Chem. Soc.*, 1948, **70**, 1153.

Tiglic Aldehyde (1 : 2-*Dimethylacrolein*)

$\quad\quad CH_3$
$CH_3{\cdot}CH{:}C{\cdot}CHO$

C_5H_8O MW, 84

Liq. with penetrating odour. B.p. 116·5–117·5°/738 mm., 63·2–65·0°/119 mm. D_4^{20} 0·8710. n_D^{20} 1·4475. Sol. 40–50 parts H_2O. Misc. with EtOH, Et_2O. Oxidises in air. Forms cryst. bisulphite comp.

Oxime: cryst. from Et_2O. M.p. 43°. B.p. 66–7°/10 mm.

Semicarbazone: needles from EtOH. M.p. 219° (234°).

Phenylhydrazone: needles from petrol. M.p. 92–4°. B.p. 163-8°/17 mm., 155–60°/9 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Mod. sol. petrol, pet. ether.

p-*Nitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 181°.

2 : 4-*Dinitrophenylhydrazone*: red cryst. from AcOEt–Me_2CO. M.p. 215·5–217·5° (222°).

Pummerer, Reindel, *Ber.*, 1933, **66**, 335.
Shepard, Johnson, *J. Am. Chem. Soc.*, 1932, **54**, 4390.
Auwers, Krender, *Ber.*, 1925, **58**, 1978.
Grignard, Ablemann, *Bull. soc. chim.*, 1910, **7**, 643.
Bernhauer, Skudrzyk, *J. prakt. Chem.*, 1940, **155**, 310.

Tigliceric Acid (1 : 2-*Dimethylglyceric acid*)

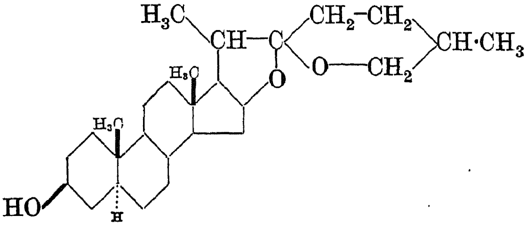
$\quad\quad\quad\quad\quad CH_3$
$CH_3{\cdot}CH(OH){\cdot}C(OH){\cdot}COOH$

$C_5H_{10}O_4$ MW, 134

Stereoisomeric with angliceric acid. Prisms from Et_2O. M.p. 88°. Very sol. H_2O, EtOH, Me_2CO. Spar. sol. cold Et_2O. Insol. $CHCl_3$, C_6H_6, ligroin. Most salts sol. H_2O.

Fittig, Penschuck, *Ann.*, 1894, **283**, 109.

Tigogenin



$C_{27}H_{44}O_3$ MW, 416

Occurs in *Digitalis purpurea*, *D. lanata*, *Chlorugalum pomeridianum*. Cryst. + $1H_2O$ from EtOH. M.p. 205–6°. Sol. Et_2O, Me_2CO, pet. ether. $[\alpha]_D^{18}$ − 67·2° in $CHCl_3$.

Acetyl: plates from MeOH. M.p. 204–6°. $[\alpha]_D^{18}$ − 74·4° in $CHCl_3$.

Benzoyl: cryst. from MeOH–$CHCl_3$. M.p. 230–3°. $[\alpha]_{5461}^{22.5}$ − 68° in $CHCl_3$.

o-*Bromobenzoyl*: cryst. from EtOH–$CHCl_3$. M.p. 210–12°. $[\alpha]_{5461}^{27}$ − 56° in $CHCl_3$.

Jacobs, Fleck, *J. Biol. Chem.*, 1930, **88**, 545.
Liang, Noller, *J. Am. Chem. Soc.*, 1935, **57**, 526.
Tschesche, Hagedorn, *Ber.*, 1935, **68**, 1416, 2247.
Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd Ed., p. 591, (Reinhold Publishing Corporation, New York).

Tigonin

$C_{56}H_{92}O_{27}$ MW, 1196

Glycoside present in leaves of *Digitalis lanata*. Amorph. from EtOH. Sinters at 220°. M.p. 260°. Absorbs $2H_2O$ in moist air. Forms add. comp. with cholesterol. Hyd. ⟶ tigogenin + galactose + glucose + xylose.

Cholesterol add. comp.: needles from MeOH. Decomp. above 200°.

Tschesche, *Ber.*, 1936, **69**, 1665.

Tin diethyl

$Sn(C_2H_5)_2$

$C_4H_{10}Sn$ MW, 177

Yellow oil. Decomp. on dist. ⟶ Sn + tin tetra-ethyl. Sol. EtOH, Et_2O, ligroin, C_6H_6. Insol. H_2O. Oxidises in air ⟶ diethyl tin oxide. Cl ⟶ Et_2SnCl_2. Reduces $AgNO_3$.

Pfeiffer, *Ber.*, 1911, **44**, 1270.
Harada, *Chem. Abstracts*, 1939, **33**, 5357.

Tin tetra-*n*-amyl

$Sn(CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3)_4$

$C_{20}H_{44}Sn$ MW, 403

Colourless stable liq. B.p. 181°/10 mm. D_4^{20} 1·0206. n_D^{20} 1·4720. Heat of comb. 3384 Cal.

Jones, Evans, Gulwell, Griffiths, *J. Chem. Soc.*, 1935, 41.

Tin tetra-*active*-amyl

$Sn(CH_2{\cdot}CH(CH_3){\cdot}CH_2{\cdot}CH_3)_4$

$C_{20}H_{44}Sn$ MW, 403

Colourless stable liq. B.p. 174°/10 mm. D_4^{20} 1·0222. n_D^{20} 1·4730.

Jones, Evans, Gulwell, Griffiths, *J. Chem. Soc.*, 1935, 41.

Tin tetrabenzyl

$Sn(CH_2{\cdot}C_6H_5)_4$

$C_{28}H_{28}Sn$ MW, 483

Prisms from pet. ether. M.p. 42–3°. Very sol. most org. solvents. Spar. sol. pet. ether. Oxidises in air ⟶ benzaldehyde.

Smith, Kipping, *J. Chem. Soc.*, 1912, **101**, 2559.

Tin tetra-*n*-butyl

$Sn(CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3)_4$

$C_{16}H_{36}Sn$ MW, 347

Colourless stable liq. B.p. 145°/10 mm. D_4^{20} 1·0572. n_D^{20} 1·4730. Heat of comb. 2773 Cal.

Jones, Evans, Gulwell, Griffiths, *J. Chem. Soc.*, 1935, 41.

Tin tetracyclohexyl

$Sn\left[-CH\begin{matrix}<CH_2-CH_2>\\ <CH_2-CH_2>\end{matrix}CH_2\right]_4$

$C_{24}H_{44}Sn$ MW, 451

Plates from C_6H_6. M.p. 263–4°. Very sol. hot C_6H_6. Spar. sol. hot EtOH, warm Et_2O. Insol. H_2O.

Krause, Pohland, *Ber.*, 1924, **57**, 535.

Tin tetraethyl

$Sn(C_2H_5)_4$

$C_8H_{20}Sn$ MW, 235

Colourless stable liq. F.p. − 112°. B.p. 175°, 73°/10 mm. $D_4^{19.7}$ 1·1988. $n_D^{19.7}$ 1·4724. Sol. Et_2O. Insol. H_2O. Heat of comb. 1521 Cal.

Emmert, Eller, *Ber.*, 1911, **44**, 2331.
Pfeiffer, Schnurmann, *Ber.*, 1904, **37**, 320.
Korsching, *Chem. Abstracts*, 1947, **41**, 1902.
Harada, *Chem. Abstracts*, 1939, **33**, 5357.

Tin tetraisoamyl

$Sn(CH_2{\cdot}CH_2{\cdot}CH(CH_3)_2)_4$

$C_{20}H_{44}Sn$ MW, 403

Liq. B.p. 188°/24 mm. $D_4^{19.6}$ 1·0353. n_D^{16} 1·4724.

Krause, *Ber.*, 1918, **51**, 1456.

Tin tetraisobutyl

$Sn(CH_2{\cdot}CH(CH_3)_2)_4$

$C_{16}H_{36}Sn$ MW, 347

Needles. M.p. – 13°. B.p. 143°/16·5 mm. D_4^{23} 1·0540. n_D^{23} 1·4742.

Grüttner, Krause, *Ber.*, 1917, **50**, 1806.

Tin tetramethyl

$Sn(CH_3)_4$

$C_4H_{12}Sn$ MW, 179

Liq. with ethereal odour. B.p. 78°. $D_4^{25\cdot5}$ 1·29136. n_D^{25} 1·4386. Insol. H_2O. Reduces alc. $AgNO_3$.

Kraus, Callis, U.S.P., 1,639,947, (*Chem. Abstracts*, 1927, **21**, 3180).
Cahours, *Ann.*, 1860, **114**, 372.
Korsching, *Zeitschrift für Naturforschung*, 1946, **1**, 219.

Tin tetraphenyl

$Sn(C_6H_5)_4$

$C_{24}H_{20}Sn$ MW, 427

Prisms from $CHCl_3$ or dipropylamine, needles from Py. M.p. 225·7°. Very sol. at the boil in $CHCl_3$, AcOH, CS_2, Py, C_6H_6. Spar. sol. EtOH, Et_2O. Insol. pet. ether.

Chambers, Scherer, *J. Am. Chem. Soc.*, 1926, **48**, 1054.
Emmert, Eller, *Ber.*, 1911, **44**, 2331.
Pope, Peachey, *Chem. News*, 1904, **89**, 20.

Tin tetrapropyl

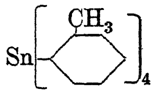
$Sn(CH_2{\cdot}CH_2{\cdot}CH_3)_4$

$C_{12}H_{28}Sn$ MW, 291

Colourless stable liq. B.p. 228°, 112°/10 mm. Heat of comb. 2163 Cal.

Pfeiffer, *Z. anorg. Chem.*, 1910, **68**, 121.

Tin tetra-*o*-tolyl

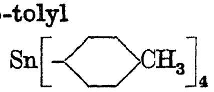


$C_{28}H_{28}Sn$ MW, 483

Cryst. from C_6H_6. M.p. 214–15°. Very sol. C_6H_6. Sol. Et_2O. Insol. EtOH.

Krause, Becker, *Ber.*, 1920, **53**, 185.
Krause, Schmitz, *Ber.*, 1919, **52**, 2158.

Tin tetra-*p*-tolyl



$C_{28}H_{28}Sn$ MW, 483

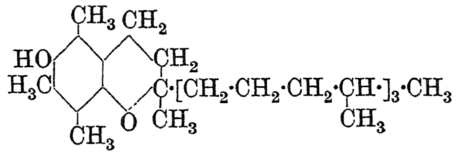
Needles from Py. M.p. 230°. Very sol. hot AcOEt, $CHCl_3$, CS_2, C_6H_6, hot Py. Spar. sol. Et_2O, EtOH, hot AcOH.

Pfeiffer, *Z. anorg. Chem.*, 1910, **68**, 122.

T.N.T.
See 2 : 4 : 6-Trinitrotoluene.

Tobias Acid.
See 2-Naphthylamine-1-sulphonic Acid.

α-Tocopherol (*Anti-sterility vitamin, vitamin E*)



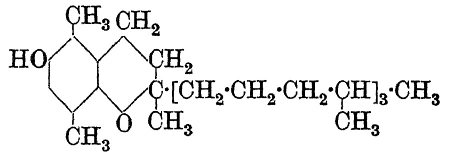
$C_{29}H_{50}O_2$ MW, 430

α-Tocopherol is a constituent of wheat-germ oil. Concentrates with high activity can be obtained from hempseed oil, cotton-seed oil, crude olive oil, green leafy vegetables, many seeds and muscle tissues. Viscous oil. B.p. 140°/10^{-6} mm. Cryst. from MeOH at – 35°. M.p. 2·5–3·5°. Sol. most solvents. Shows absorption maxima at 292 mμ and 298 mμ. Thermostable. Resistant to light, acids, alkalis and mild oxidation.

Acetyl: m.p. 26·5–27·5°.
3 : 5-*Dinitrobenzoyl*: m.p. 66–7°.
Bromocamphorsulphonyl: m.p. 52°. $[\alpha]_D$ + 40°.
Allophanate: cryst. from MeOH. M.p. 158–60°.
p-*Nitrophenylurethane*: cryst. from MeOH. M.p. 129–31°.

Evans, Emerson, Emerson, *J. Biol. Chem.*, 1936, **113**, 319.
Karrer *et al.*, *Helv. Chim. Acta*, 1938, **21**, 520, 820.
Bergel *et al.*, *J. Chem. Soc.*, 1938, 1382.
Smith, Ungnade, Pritchard, *Science*, 1938, **88**, 37.

β-Tocopherol (*Vitamin E*)



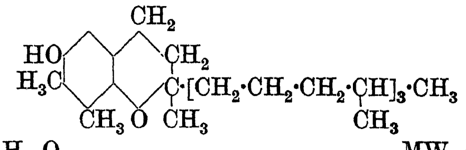
$C_{28}H_{48}O_2$ MW, 416

Occurs in wheat-germ oil. Viscous oil. Sol. most fat-soluble solvents. Shows absorption maximum at 297 mμ. Thermostable. Resistant to acids and alkalis.

p-*Nitrobenzoyl*: m.p. 38–40°.
3 : 5-*Dinitrobenzoyl*: m.p. 86–7°.
Allophanate: m.p. 138–9°.
p-*Nitrophenylurethane*: m.p. 90°.

Karrer, Fritzsche, *Helv. Chim. Acta*, 1938, **21**, 1234; 1939, **22**, 260.

γ-Tocopherol (*Vitamin E*)



$C_{28}H_{48}O$ MW, 416

Occurs in cotton-seed oil. Viscous oil. Cryst. from MeOH at − 35°. M.p. − 3° to − 2°. Sol. most fat-soluble solvents. Absorption maximum at 298 mμ. Thermostable. Resistant to acids and alkalis.

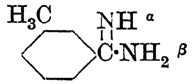
Allophanate : m.p. 136–8°.

p-*Nitrophenylurethane* : m.p. 119–21°.

3 : 5-*Dinitrophenylurethane* : m.p. 143–5°.

Karrer, Fritzsche, *Helv. Chim. Acta*, 1939, **22**, 260.

***m*-Tolamidine** (m-*Tolenylamidine*, m-*guanyltoluene*)



$C_8H_{10}N_2$ MW, 134

B,HCl : m.p. 185·5°.

Ekeley, Tieszen, Ronzio, *J. Am. Chem. Soc.*, 1935, **57**, 381.

***p*-Tolamidine** (p-*Tolenylamidine*, p-*guanyltoluene*).

Plates from C_6H_6. M.p. 101–2°.

B,HCl : prisms + $\frac{1}{2}H_2O$ from H_2O. M.p. 213°.

B_2,H_2SO_4 : prisms + $2H_2O$. Does not melt below 240°.

B,H_2SO_4 : plates from EtOH–Et_2O. M.p. 240–1°.

B,HNO_2 : needles. M.p. 133°.

B,HNO_3 : prisms from EtOH, m.p. 154°; cryst. + H_2O from H_2O, m.p. 95°.

Carbonate : m.p. 118–22°.

B_2,H_2PtCl_6 : yellow needles. M.p. 225° (198°).

Picrate : m.p. 219° decomp.

α : β-N-*Di-Me* : $C_{10}H_{14}N_2$. MW, 162. *B,HCl* : needles from H_2O. M.p. 200°. B_2,H_2PtCl_6 : cryst. + $2H_2O$. M.p. 95°.

β-N-*Et* : $C_{10}H_{14}N_2$. MW, 162. *B,HCl* : needles. M.p. 212°. B_2,H_2PtCl_6 : needles + $4H_2O$. M.p. 65°.

β-N-*Phenyl* : $C_{14}H_{14}N_2$. MW, 210. Cryst. from C_6H_6. M.p. 149°. Very sol. EtOH. Spar. sol. C_6H_6, hot ligroin.

β-N-*Acetyl* : plates from EtOH–AcOEt. M.p. 108°.

β-N-p-*Toluyl* : needles from EtOH. M.p. 145°.

Bernton, *Chem. Zentr.*, 1919, III, 329.
Glock, *Ber.*, 1888, **21**, 2653.
Kirsanov, Polyakova, *Chem. Abstracts*, 1936, **30**, 8156.

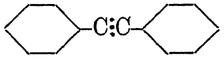
α-Tolamidine.

See Phenylacetamidine.

Tolamine.

See Chloramine-T.

Tolane (sym.-*Diphenylacetylene*)



$C_{14}H_{10}$ MW, 178

Plates or columns from EtOH. M.p. 62·5°. Dist. without decomp. Very sol. Et_2O, hot EtOH. Spar. sol. cold EtOH. Heat of comb. C_p 1738·2 Cal., C_v 1736·7 Cal. Latent heat of fusion 28·7 cal./gm. Ox. ⟶ benzoic acid. Conc. H_2SO_4 at 60° ⟶ deoxybenzoin. Red. ⟶ isostilbene + stilbene + dibenzyl.

$C_{14}H_{10},C_6H_3(NO_2)_3$-1 : 3 : 5 : yellow plates. M.p. 96°.

Picrate : yellow plates. M.p. 111°.

Weissberger, *J. Chem. Soc.*, 1935, 855.
Fittig, *Ann.*, 1873, **168**, 74.
Schlenk, Bergmann, *Ann.*, 1928, **463**, 76.
Smith, Falkof, *Organic Syntheses*, 1942, XXII, 50.

Tolane dibromide (α : β-*Dibromostilbene*, 1 : 2-*dibromo*-1 : 2-*diphenylethylene*)

$C_6H_5·CBr{:}CBr·C_6H_5$

$C_{14}H_{10}Br_2$ MW, 338

Exists in two forms, probably stereoisomeric.

α-.

Plates or needles. M.p. 205–6°. Spar. sol. Et_2O, hot EtOH. Difficultly volatile in steam. Heat with H_2O at 170–80° ⟶ β-form. Hot alc. KOH ⟶ tolane.

β-.

Needles. M.p. 64°. Very sol. Et_2O, EtOH. Difficultly volatile in steam. Heat with H_2O at 170–80° ⟶ α-form. Hot alc. KOH ⟶ tolane.

Staudinger, *Ber.*, 1916, **49**, 1972.
Limpricht, Schwanert, *Ber.*, 1871, **4**, 379.

Tolane dichloride (α : β-*Dichlorostilbene*, 1 : 2-*dichloro*-1 : 2-*diphenylethylene*)

$C_6H_5·CCl{:}CCl·C_6H_5$

$C_{14}H_{10}Cl_2$ MW, 249

Exists in two forms, probably stereoisomeric.

α-.

Plates from EtOH. M.p. 140–2° (153°). B.p. 183°/18 mm. Sol. 130 parts EtOH at 24°. Very sol. Et_2O. Heat. ⟶ β-form. HI(+ P) ⟶ dibenzyl. NaHg or alc. KOH ⟶ tolane.

β-.

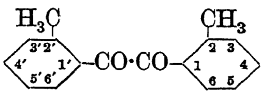
Needles. M.p. 63°. B.p. 178°/18 mm. Sol. 10 parts EtOH at 24°. Very sol. Et_2O. Heat. ⟶ α-form.

Staudinger, *Ber.*, 1916, **49**, 1971.
Loeb, *Ber.*, 1903, **36**, 3060.
Limpricht, Schwanert, *Ber.*, 1871, **4**, 379.
Il'ichev, *Chem. Abstracts*, 1949, **43**, 1755.

Tolidine.

See Diaminodimethyldiphenyl.

***o*-Tolil** (*Di*-o-*tolyl diketone*, 2 : 2′-*dimethylbenzil*)



$C_{16}H_{14}O_2$ MW, 238

Yellow needles from EtOH. M.p. 92–4°.

Fuson, Rachlin, *J. Am. Chem. Soc.*, 1946, **68**, 343.

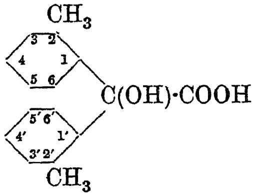
***m*-Tolil** (*Di*-m-*tolyl diketone*, 3 : 3′-*dimethylbenzil*).
Yellowish-white needles from EtOH. M.p. 103°.

Ekecrantz, Ahlquist, *Chem. Zentr.*, 1908, II, 1689.

***p*-Tolil** (*Di*-p-*tolyl diketone*, 4 : 4′-*dimethylbenzil*).
Yellow plates from EtOH. M.p. 104–5°. Sol. Et_2O, AcOH, C_6H_6. Alc. sol. + KOH ⟶ violet col.
Dioxime: *syn*-: α-*p*-tolildioxime. Plates or needles. M.p. 217°. Spar. sol. EtOH, Et_2O, AcOH. *Diacetyl*: prisms. M.p. 133–4°. *Anti*-: β-*p*-tolildioxime. Needles. M.p. 225°. Very sol. EtOH. *Diacetyl*: m.p. 144°. *Amphi*-: γ-*p*-tolildioxime. Needles + $1CHCl_3$ from $CHCl_3$. Sinters about 200°. M.p. 229–32°. Long heat. at 200° ⟶ β-dioxime.
Monohydrazone: needles from EtOH. M.p. 139–40°.
Dihydrazone: needles from EtOH. M.p. 137°.

Vorländer, *Ber.*, 1911, **44**, 2461.
Tschugajew, Spiro, *Ber.*, 1908, **41**, 2221.
Klein, *J. Am. Chem. Soc.*, 1941, **63**, 1474.

***o*-Tolilic Acid** (2 : 2′-*Dimethylbenzilic acid, di*-o-*tolylglycollic acid*, α-*hydroxy-di*-o-*tolylacetic acid*)



$C_{16}H_{16}O_3$ MW, 256

Cryst. from C_6H_6. M.p. 162–3°. H_2SO_4 ⟶ purple col.
Et ester: m.p. 55°.

Fuson, Rachlin, *J. Am. Chem. Soc.*, 1946, **68**, 343.

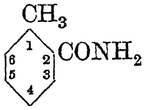
***p*-Tolilic Acid** (4 : 4′-*Dimethylbenzilic acid, di*-p-*tolylglycollic acid*, α-*hydroxy-di*-p-*tolylacetic acid*).
Colourless needles from H_2O. M.p. 135°. Sol. most org. solvents. Spar. sol. ligroin. Insol. cold H_2O. H_2SO_4 ⟶ purple col.
Me ester: $C_{17}H_{18}O_3$. MW, 270. Needles from EtOH. M.p. 82°. Very sol. EtOH, Et_2O, $CHCl_3$.
Anhydride: $C_{32}H_{30}O_5$. MW, 494. Prisms from EtOH. M.p. 164°.
Acetyl: cryst. from pet. ether. M.p. 92°.

Gattermann, *Ann.*, 1906, **347**, 364.
Gisiger, *Ber.*, 1906, **39**, 3589.

Toliminazole.
See Methylbenziminazole.

Tolualdehyde.
See Toluic Aldehyde.

***o*-Toluamide**



C_8H_9ON MW, 135

Plates from H_2O, needles from EtOH. M.p. 147° (138°). Sol. hot H_2O, EtOH. Spar. sol. Et_2O, C_6H_6. NaHg in slightly acid sol. ⟶ *o*-tolylcarbinol. NaHg in slightly alk. sol. ⟶ dihydro-*o*-toluamide. EtOH + H_2SO_4 ⟶ ethyl *o*-toluate.
N-*Me*: $C_9H_{11}ON$. MW, 149. Needles from H_2O. M.p. 75°.
N-*Di-Me*: $C_{10}H_{13}ON$. MW, 163. Liq. B.p. 147°/18 mm. D^{25} 1·033.
N-*Benzoyl*: needles from EtOH.Aq. M.p. 158–9°.
N-*Xanthyl*: cryst. from 65% aq. dioxan. M.p. 199–200°.
N-*Sulphanilyl*: m.p. 176°.

Noller, *Organic Syntheses*, 1933, XIII, 94.

***m*-Toluamide.**
Needles from Et_2O. M.p. 97° (93°). Spar. sol. Et_2O, C_6H_6.
N-*Me*: m.p. 44·5–45°. Sol. H_2O, EtOH, Et_2O.
N-*Di-Me*: b.p. 148°/12 mm. D^{15} 1·043.
N-*Benzyl*: m.p. 75°.
N-*Sulphanilyl*: m.p. 160°.

Remsen, Reid, *Am. Chem. J.*, 1899, **21**, 289.

***p*-Toluamide.**
Needles or plates from H_2O, needles from C_6H_6. M.p. 155° (165°). Very sol. hot H_2O, EtOH, Et_2O. Spar. sol. cold H_2O, $CHCl_3$, C_6H_6.
N-*Me*: plates from H_2O. M.p. 145–145·5°.
N-*Di-Me*: cryst. from EtOH. M.p. 41°. B.p. 156°/10 mm. Sol. H_2O, EtOH.
N-*Et*: $C_{10}H_{13}ON$. MW, 163. Needles from H_2O. M.p. 90°.
N-*Benzyl*: m.p. 133°.
N-*Acetyl*: prisms from EtOH.Aq. M.p. 147°.
N-*Benzoyl*: needles from EtOH.Aq. M.p. 119°.
N-*Xanthyl*: cryst. from 65% aq. dioxan. M.p. 224°.
N-*Sulphanilyl*: m.p. 144°.

McMaster, Langreck, *J. Am. Chem. Soc.*, 1917, **39**, 106.

Tolubenzyl cyanide.
See under Tolylacetic Acid.

Toluene (*Methylbenzene*)

$C_6H_5{\cdot}CH_3$

C_7H_8 MW, 92

Constituent of many mineral oils. Important constituent of coal tar oil. Liq. F.p. — 95·0°. B.p. 110·6°, 102·6°/600 mm., 79·7°/288 mm., 14·5°/14·56 mm. Vap. press. at 0° 6·5 mm., at 20° 22 mm., at 50° 93·5 mm., at 60° 141·5 mm., at 70° 203 mm., at 80° 292·5 mm., at 100° 558 mm. Very slightly sol. H_2O. D_4^0 0·8845, D_4^{20} 0·866. n_D^{20} 1·49613 (1·49693). Heat of comb. C_p 937·4 Cal., C_v 938·5 Cal., C_p (vapour) 955·68 Cal. Crit. temp. 320·8°. Crit. press. 41·6 atm. Specific heat 0·404 cal./gm. at 25°. Latent heat of fusion 0·0016 Cal. Latent heat of vap. 0·00094 Cal. at 110·2°. Forms constant b.p. mixture with acetic acid, b.p. 104–104·2°.

Picrate: pale yellow cryst. M.p. 88·2°.

Perkin, *J. Chem. Soc.*, 1896, **69**, 1191.

Tolueneazoanisole.

See under Hydroxymethylazobenzene.

Toluenazobenzoic Acid.

See Methylazobenzene-carboxylic Acid.

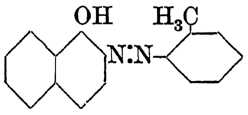
Tolueneazocresol.

See Hydroxydimethylazobenzene.

5-*p*-Tolueneazo-ψ-cumene.

See 2 : 4 : 5 : 4′-Tetramethylazobenzene.

2-*o*-Tolueneazo-1-naphthol (1 : 2-*Naphthoquinone*-2-o-*tolylhydrazone*)



$C_{17}H_{14}ON_2$ MW, 262

Red leaflets. M.p. 156°. Sol. most org. solvents.

Me ether: $C_{18}H_{16}ON_2$. MW, 276. Orange-yellow needles from EtOH. M.p. 84–5°. Sol. Et_2O, C_6H_6, $CHCl_3$. Less sol. ligroin. Conc. H_2SO_4 ⟶ red sol.

Et ether: $C_{19}H_{18}ON_2$. MW, 290. Yellow needles from EtOH. M.p. 51°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Less sol. pet. ether. Conc. H_2SO_4 ⟶ ruby-red sol.

Zincke, Rathgen, *Ber.*, 1886, **19**, 2492.

Charrier, Ferreri, *Gazz. chim. ital.*, 1914, **44**, ii, 234.

2-*m*-Tolueneazo-1-naphthol (1 : 2-*Naphthoquinone*-2-m-*tolylhydrazone*).

Red needles from EtOH. M.p. 117–18°. Very sol. C_6H_6, $CHCl_3$. Sol. Et_2O. Spar. sol. pet. ether. Insol. cold EtOH. Conc. H_2SO_4 ⟶ red sol.

Me ether: yellow leaflets from EtOH. M.p. 49–50°. Sol. most org. solvents. Conc. H_2SO_4 ⟶ red sol.

Et ether: orange-red prisms from EtOH. M.p. 22°. Very sol. Et_2O, C_6H_6, $CHCl_3$. Sol. EtOH. Conc. H_2SO_4 ⟶ ruby-red sol.

Charrier, Ferreri, *Gazz. chim. ital.*, 1914, **44**, ii, 235.

2-*p*-Tolueneazo-1-naphthol (1 : 2-*Naphthoquinone*-2-p-*tolylhydrazone*).

Red needles from EtOH. M.p. 145°. Sol. EtOH, C_6H_6, AcOH. Spar. sol. pet. ether. Insol. alkalis. Conc. H_2SO_4 ⟶ red sol.

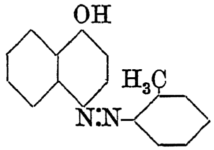
Me ether: orange-red needles from EtOH. M.p. 77–8°. Sol. Et_2O, boiling EtOH, C_6H_6, $CHCl_3$. Spar. sol. pet. ether. Conc. H_2SO_4 ⟶ ruby-red sol.

Et ether: orange-yellow needles from EtOH. M.p. 51°. Sol. most org. solvents. Conc. H_2SO_4 ⟶ ruby-red sol.

Charrier, Ferreri, *Gazz. chim. ital.*, 1914, **44**, ii, 235.

Charrier, Casale, *Gazz. chim. ital.*, 1914, **44**, i, 611.

4-*o*-Tolueneazo-1-naphthol (1 : 4-*Naphthoquinone*-o-*tolylhydrazone*)



$C_{17}H_{14}ON_2$ MW, 262

Red needles from 66% EtOH. M.p. 144–6°. Sol. EtOH, C_6H_6, AcOH. Less sol. pet. ether.

Me ether: $C_{18}H_{16}ON_2$. MW, 276. Reddish-brown needles. M.p. 93°. Sol. usual org. solvents.

Et ether: $C_{19}H_{18}ON_2$. MW, 290. Red plates from EtOH. M.p. 94°.

Zincke, Rathgen, *Ber.*, 1886, **19**, 2488.

4-*p*-Tolueneazo-1-naphthol (1 : 4-*Naphthoquinone*-p-*tolylhydrazone*).

Dark red cryst. from $PhNO_2$. M.p. 209–10°. Sol. Me_2CO, $PhNO_2$, aniline. Spar. sol. EtOH, C_6H_6, AcOH.

Me ether: cryst. M.p. 103–4°.

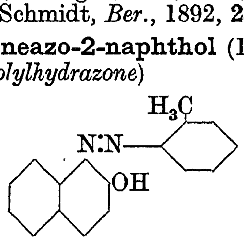
Et ether: red needles from EtOH. M.p. 126–7°.

Acetyl: yellowish needles from pet. ether. M.p. 101–2°.

Zincke, Rathgen, *Ber.*, 1886, **19**, 2488.

Witt, Schmidt, *Ber.*, 1892, **25**, 1019.

1-*o*-Tolueneazo-2-naphthol (1 : 2-*Naphthoquinone*-1-o-*tolylhydrazone*)



$C_{17}H_{14}ON_2$ MW, 262

Red needles from AcOH. M.p. 131° (130°).

Me ether: $C_{18}H_{16}ON_2$. MW, 276. Red plates from EtOH. M.p. 58°. Sol. most org. solvents. Conc. H_2SO_4 ⟶ red sol. *B,2HNO₃*: green needles. M.p. 71° decomp. Sol. EtOH, Et_2O. Mod. sol. $CHCl_3$. Spar. sol. C_6H_6, ligroin.

Et ether: $C_{19}H_{18}ON_2$. MW, 290. Red needles from EtOH. M.p. 36°. Sol. most org. solvents. *B,2HNO₃*: green leaflets. M.p. 62–3°. Mod. sol. EtOH, $CHCl_3$. Spar. sol. C_6H_6, ligroin.

Norman, *J. Chem. Soc.*, 1912, **101**, 1920.
Charrier, Ferreri, *Gazz. chim. ital.*, 1912, **42**, ii, 124; 1913, **43**, i, 549.

1-m-Tolueneazo-2-naphthol (1:2-*Naphthoquinone*-1-m-*tolylhydrazone*).

Red needles from C_6H_6. M.p. 141° (137°). Sol. most org. solvents. Conc. $H_2SO_4 \longrightarrow$ violet sol.

Me ether: red plates from EtOH. M.p. 81°. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Spar. sol. pet. ether. Conc. acids $\longrightarrow$ red sols. *B,2HNO₃*: green leaflets. M.p. 72°. Sol. EtOH, $CHCl_3$. Less sol. Et_2O, C_6H_6. Spar. sol. pet. ether.

Et ether: red leaflets from ligroin. M.p. 84°. Sol. most org. solvents. *B,2HNO₃*: green leaflets. M.p. 84° decomp. Sol. EtOH, $CHCl_3$. Insol. C_6H_6.

Norman, *J. Chem. Soc.*, 1912, **101**, 1920.
Charrier, Ferreri, *Gazz. chim. ital.*, 1912, **42**, ii, 124; 1913, **43**, i, 549.

1-p-Tolueneazo-2-naphthol (1:2-*Naphthoquinone*-1-p-*tolylhydrazone*).

Red needles or plates from AcOH. M.p. 134–5°. Sol. EtOH, Me_2CO, C_6H_6, AcOH.

Me ether: red plates from EtOH. M.p. 68°. Sol. most org. solvents. Conc. $H_2SO_4 \longrightarrow$ ruby-red sol. *B,2HNO₃*: red needles. M.p. 77° decomp. Sol. warm $CHCl_3$. Spar. sol. Et_2O. Insol. C_6H_6.

Et ether: red needles from EtOH. M.p. 51°. Very sol. Et_2O, C_6H_6, $CHCl_3$. Less sol. EtOH, pet. ether. *B,2HNO₃*: red leaflets. M.p. 94° decomp. Mod. sol. EtOH, $CHCl_3$. Less sol. Et_2O, C_6H_6. Insol. ligroin.

Norman, *J. Chem. Soc.*, 1912, **101**, 1920.
Charrier, Ferreri, *Gazz. chim. ital.*, 1912, **42**, ii, 124; 1913, **43**, i, 549.

Tolueneazophenetole.
See under Hydroxy-methylazobenzene.

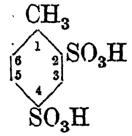
Tolueneazophenol.
See Hydroxy-methylazobenzene.

Tolueneazotoluidine.
See Aminodimethylazobenzene.

Tolueneazoxylenol.
See 2-Hydroxy-3 : 5 : 4′-trimethylazobenzene.

Toluene-dicarboxylic Acid.
See Methylphthalic Acid, Methylisophthalic Acid *and* Methylterephthalic Acid.

Toluene-2 : 4-disulphonic Acid



$C_7H_8O_6S_2$ MW, 252

Thick oil. Ox. of K salt $\longrightarrow$ 2 : 4-disulphobenzoic acid. Fuse K salt with H·COONa $\longrightarrow$ 4-methylisophthalic acid.

p-*Toluidine salt*: m.p. 277° decomp.

Difluoride: $C_7H_6O_4F_2S_2$. MW, 256. Plates from petrol. M.p. 87–8°. Sol. Me_2CO, AcOEt, $PhNO_2$. Mod. sol. EtOH, Et_2O, CS_2. Spar. sol. pet. ether.

Dichloride: $C_7H_6O_4Cl_2S_2$. MW, 289. Prisms from Et_2O. M.p. 56°. Very sol. Et_2O, C_6H_6.

Dibromide: $C_7H_6O_4Br_2S_2$. MW, 378. Cryst. from Et_2O. M.p. 78°.

Diamide: $C_7H_{10}O_4N_2S_2$. MW, 250. Prisms from EtOH or H_2O. M.p. 190–1°. Sol. warm H_2O. Very sol. EtOH, NH_4OH.

Dianilide: prisms from EtOH.Aq., needles from C_6H_6, plates from Me_2CO. M.p. 189°.

Di-o-toluidide: cryst. M.p. 170–1°.

Di-m-toluidide: cryst. M.p. 138°.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 38.
Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 757.
Gnehm, Forrer, *Ber.*, 1877, **10**, 542.

Toluene-2 : 5-disulphonic Acid.

Dichloride: prisms or plates from C_6H_6 or C_6H_6–pet. ether. M.p. 98°. Very sol. AcOEt, $CHCl_3$, CS_2, C_6H_6. Spar. sol. pet. ether.

Diamide: prisms. M.p. 224°. Spar. sol. H_2O.

Dianilide: needles from C_6H_6 or EtOH.Aq. M.p. 178°.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 757.

Toluene-2 : 6-disulphonic Acid.

Dichloride: prisms from pet. ether or C_6H_6. M.p. 88°. Very sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. pet. ether.

Diamide: needles. Does not melt below 260°. Mod. sol. EtOH. Spar. sol. H_2O.

Dianilide: prisms from EtOH.Aq. or C_6H_6. M.p. 162°.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 771.

Toluene-3 : 4-disulphonic Acid.

Forms cryst. spar. sol. Ba salt.

Dichloride: scales from C_6H_6–pet. ether, m.p. 111°; cryst. + $\frac{1}{2}C_6H_6$ from C_6H_6, m.p. 70–80°.

Diamide: m.p. 235–9° (slight decomp.). Very sol. H_2O, EtOH.

Dianilide: plates from Me_2CO, prisms from EtOH. M.p. 190°.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 751.
Klason, *Ber.*, 1887, **20**, 356.

Toluene-3 : 5-disulphonic Acid.

Needles. Very sol. H_2O, EtOH.

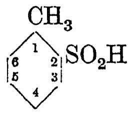
Dichloride: prisms from Et_2O. M.p. 95°. Very sol. Et_2O. Spar. sol. pet. ether.

Diamide: prisms from H_2O. M.p. 216°. Sol. hot H_2O, EtOH. Spar. sol. cold H_2O.

Dianilide: scales from C_6H_6 or EtOH.Aq. M.p. 153°.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 734, 738.
Richter, *Ann.*, 1885, **230**, 326.

Toluene-*o*-sulphinic Acid



$C_7H_8O_2S$ MW, 156

Needles. M.p. 80°. Very sol. most org. solvents. Easily decomp. Salts very sol. H_2O.

Höchst, D.R.P., 224,019, (*Chem. Zentr.*, 1910, II, 513).
Gattermann, *Ber.*, 1899, **32**, 1140.

Toluene-*m*-sulphinic Acid.

Unstable oil with strong bleaching properties.

Troeger, Hille, *J. prakt. Chem.*, 1905, **71**, 207.

Toluene-*p*-sulphinic Acid.

d-.

Et ester: $C_9H_{12}O_2S$. MW, 184. B.p. 60–1°/high vacuum. n_D^{25} 1·5309. $[\alpha]_{5461}^{25}$ + 0·92°.

l-.

Et ester: b.p. 63°/high vacuum. n_D^{25} 1·5309. $[\alpha]_{5461}^{25}$ − 6·72°.

Butyl ester: b.p. 84°/less than 0·1 mm. D_4^{25} 1·066. n_D^{25} 1·5195.

Anilide: plates from EtOH.Aq. M.p. 134°. B.p. 75–6°/less than 0·1 mm. $[\alpha]_{5461}^{17}$ − 1·1° in $CHCl_3$.

dl-.

Dark plates or needles from H_2O. M.p. 85°. Very sol. EtOH, Et_2O. Spar. sol. cold H_2O, hot C_6H_6. Hygroscopic. Very readily oxidised to toluene-*p*-sulphonic acid. Zn + H_2SO_4 ⟶ *p*-thiocresol. Alc. MeI ⟶ methyl *p*-tolyl sulphone. Forms spar. sol. Ag salt.

NH_4 salt: needles from EtOH. M.p. 175° decomp.

Et ester: b.p. 99–104°/0·1 mm. n_D^{25} 1·5309. D_4^{25} 1·114.

Butyl ester: b.p. 90–95°/0·1 mm. D_4^{25} 1·066. n_D^{25} 1·5195.

Anhydride: $C_{14}H_{14}O_3S_2$. MW, 294. M.p. 75°. Decomp. on standing.

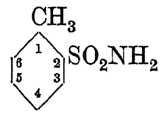
Chloride: C_7H_7OClS. MW, 174·5. Needles. M.p. 54–8°.

Amide: C_7H_9ONS. MW, 155. Needles. M.p. 120°.

Anilide: m.p. 138°.

Whitmore, Hamilton, *Organic Syntheses*, Collective Vol. I, 479.
Phillips, *J. Chem. Soc.*, 1925, 2552.
v. Braun, Kaiser, *Ber.*, 1923, **56**, 549.

Toluene-*o*-sulphonamide



$C_7H_9O_2NS$ MW, 171

Octahedra from EtOH, prisms from H_2O. M.p. 156·3°. Sol. 958 parts H_2O at 9°, 28 parts EtOH at 5°. Electrolytic oxidation of alk. metal salts ⟶ saccharin.

N-*Me*: $C_8H_{11}O_2NS$. MW, 185. Plates from C_6H_6–ligroin. M.p. 74–5°. Very sol. EtOH, Me_2CO, $CHCl_3$. Spar. sol. ligroin, hot H_2O. Insol. cold C_6H_6.

N-*Chloro deriv.*: $C_7H_8O_2NClS$. MW, 205·5. *Na salt*: prisms + $2H_2O$. Explodes (anhyd.) at 170–5°. *K salt*: prisms + H_2O. Explodes (anhyd.) about 145°.

N-*Dichloro deriv.*: $C_7H_7O_2NCl_2S$. MW, 240. Plates from $CHCl_3$–pet. ether. M.p. 33°.

N-*Bromo deriv.*: $C_7H_8O_2NBrS$. MW, 250. *Na salt*: yellow plates + $1H_2O$. Explodes (anhyd.) about 135–40°. *K salt*: yellow plates. Decomp. (anhyd.) at 130–5°.

N-*Dibromo deriv.*: $C_7H_7O_2NBr_2S$. MW, 329. Orange plates from $CHCl_3$–pet. ether. M.p. 80°. Very sol. $CHCl_3$. Spar. sol. pet. ether.

N-*Benzoyl*: *N*-*o*-toluenesulphonylbenzamide. Plates or prisms from Et_2O. M.p. 110–12°.

N-*Xanthyl*: cryst. from aq. dioxan. M.p. 182–183·5°.

Ullmann, *Encyclopedia of Technical Chemistry*, (2nd Ed., Berlin–Vienna 1928), II, 251.
Chattaway, *J. Chem. Soc.*, 1905, **87**, 152.

Toluene-*m*-sulphonamide.

Plates or cryst. aggregates from H_2O, prisms from EtOH. M.p. 108°. Sol. 130 parts H_2O at 25°. Sol. EtOH.

Griffin, *Am. Chem. J.*, 1897, **19**, 174.

Toluene-*p*-sulphonamide.

Plates + $2H_2O$ from H_2O. M.p. 105°, anhyd. 137·5°. Sol. 515 parts H_2O at 9°, 13·5 parts EtOH at 5°.

N-*Me*: $C_8H_{11}O_2NS$. MW, 185. Plates from EtOH.Aq. M.p. 78–9°.

N-*Di-Me*: $C_9H_{13}O_2NS$. MW, 199. Needles from petrol. M.p. 86–7°. Sol. AcOEt, C_6H_6. Mod. sol. Et_2O, EtOH. Spar. sol. H_2O.

N-*Et*: $C_9H_{13}O_2NS$. MW, 199. Cryst. from ligroin, plates from EtOH.Aq. M.p. 64°.

N-*Di-Et*: $C_{11}H_{17}O_2NS$. MW, 227. Cryst. from ligroin. M.p. 60°. Spar. sol. H_2O, ligroin.

N-*Propyl*: $C_{10}H_{15}O_2NS$. MW, 213. Cryst. from ligroin. M.p. 52°.

N-*Butyl*: $C_{11}H_{17}O_2NS$. MW, 227. M.p. 43°.

N-*Hexyl*: $C_{13}H_{21}O_2NS$. MW, 255. M.p. 62°.

N-*Heptyl*: $C_{14}H_{23}O_2NS$. MW, 269. M.p. 27°.

N-*Benzyl*: $C_{14}H_{15}O_2NS$. MW, 261. Prisms from EtOH. M.p. 115–16°.
N-*Dibenzyl*: $C_{21}H_{21}O_2NS$. MW, 351. Needles from MeOH. M.p. 80·8°
N-*Chloro*: *Na salt*: *see* Chloramine-T. *K salt*: plates + H_2O. Explodes (anhyd.) about 160–5°.
N-*Dichloro*: *see* Dichloramine-T.
N-*Bromo*: $C_7H_8O_2NBrS$. MW, 250. *Na salt*: yellow prisms + $3H_2O$. Decomp. (anhyd.) at 145–50°. *K salt*: yellow plates + $2H_2O$. Decomp. (anhyd.) at 145–50°.
N-*Dibromo*: $C_7H_7O_2NBr_2S$. MW, 329. Orange plates from $CHCl_3$. M.p. 104°.
N-*Acetyl*: needles. M.p. 139°.
N-*Benzoyl*: prisms or needles from EtOH. M.p. 147–50°.
N-o-*Toluyl*: m.p. 112·5°.
N-m-*Toluyl*: m.p. 132°.
N-p-*Toluyl*: m.p. 138–9°.
N-2-*Naphthoyl*: m.p. 162·5°.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 25.
Chattaway, *J. Chem. Soc.*, 1905, **87**, 152.
Wolkow, *Z. Chem.*, 1870, **323**, 578.

Toluene-α-sulphonamide (*Benzylsulphonamide*)

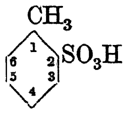
$C_6H_5{\cdot}CH_2{\cdot}SO_2NH_2$

$C_7H_9O_2NS$ MW, 171

Prisms or needles from hot H_2O, needles from EtOH. M.p. 105°. Very sol. hot H_2O, EtOH.
N-*Me*: $C_8H_{11}O_2NS$. MW, 185. Needles. M.p. 109–10°. Sol. hot H_2O, EtOH, Et_2O.
N-*Di-Me*: $C_9H_{13}O_2NS$. MW, 199. Plates from $CHCl_3$–ligroin. M.p. 101°.
N-*Et*: $C_9H_{13}O_2NS$. MW, 199. Leaflets from Et_2O–ligroin. M.p. 65–6°.
N-*Di-Et*: $C_{11}H_{17}O_2NS$. MW, 227. Leaflets from Et_2O–ligroin. M.p. 29°.
N-*Sulphanilyl*: m.p. 226–7°.

Ingold, Ingold, Shaw, *J. Chem. Soc.*, 1927, 818.
Curtius, Haas, *J. prakt. Chem.*, 1921, **102**, 102, 104, 106.
Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, **36**, 381.

Toluenesulphonbromide.
See under Toluenesulphonic Acid.
Toluenesulphonchloride.
See under Toluenesulphonic Acid.
Toluenesulphonfluoride.
See under Toluenesulphonic Acid.
Toluene-*o*-sulphonic Acid



$C_7H_8O_3S$ MW, 172

Hygroscopic plates + $2H_2O$. Heat. at 140–50° ⟶ toluene-*p*-sulphonic acid. Ox. ⟶ *p*-sulphobenzoic acid. KOH fusion ⟶ *o*-cresol + salicylic acid. Gives spar. sol. Ag, Pb salts.
Aniline salt: m.p. 218°.
l-Menthyl ester: $C_{17}H_{26}O_3S$. MW, 310. Prisms. M.p. 78°.
Phenyl ester: $C_{13}H_{12}O_3S$. MW, 248. M.p. 52°.
o-*Nitrophenyl ester*: $C_{13}H_{11}O_5NS$. MW, 293. Plates from EtOH. M.p. 131–4°.
o-*Tolyl ester*: $C_{14}H_{14}O_3S$. MW, 262. Cryst. from EtOH. M.p. 50–1°.
m-*Tolyl ester*: cryst. from EtOH. M.p. 60°.
p-*Tolyl ester*: cryst. from EtOH. M.p. 70–1°.
Fluoride: toluene-*o*-sulphonfluoride. $C_7H_7O_2FS$. MW, 174. Oil. B.p. 223–5°, 146·2°/83 mm., 133·9°/56·5 mm. n_D^{20} 1·5007.
Chloride: toluene-*o*-sulphonchloride. $C_7H_7O_2ClS$. MW, 190·5. Cryst. from $CHCl_3$. M.p. 67·5°. B.p. 126°/10 mm.
Bromide: toluene-*o*-sulphonbromide. $C_7H_7O_2BrS$. MW, 235. Cryst. M.p. 13°. B.p. 137·5–138°/10 mm.
Amide: *see* Toluene-*o*-sulphonamide.
Anilide: plates from EtOH.Aq. M.p. 136°.
o-*Toluidide*: m.p. 134°.

Terlinck, *Chem. Abstracts*, 1927, **21**, 1978.
Steinkopf, *J. prakt. Chem.*, 1927, **117**, 38.
Holleman, Caland, *Ber.*, 1911, **44**, 2505.
Ullmann, Lehner, *Ber.*, 1905, **38**, 732.

Toluene-*m*-sulphonic Acid.
Free acid is an oil.
Chloride: toluene-*m*-sulphonchloride. F.p. 11·7°. Decomp. by boiling H_2O.
Amide: *see* Toluene-*m*-sulphonamide.
Anilide: prisms from EtOH. M.p. 96°.
o-*Toluidide*: prisms and plates. M.p. 108°.
m-*Toluidide*: m.p. 103°.
p-*Toluidide*: prisms. M.p. 106°.

Holleman, Caland, *Ber.*, 1911, **44**, 2504, 2515.
Griffin, *Am. Chem. J.*, 1897, **19**, 173, 189.

Toluene-*p*-sulphonic Acid.
Hygroscopic plates + $1H_2O$ from H_2O. M.p. 92° (104–5°). Ox. ⟶ *p*-sulphobenzoic acid. KOH fusion ⟶ *p*-cresol + *p*-hydroxybenzoic acid.
Me ester: $C_8H_{10}O_3S$. MW, 186. Cryst. from Et_2O–ligroin. M.p. 28°. Very sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.
Et ester: $C_9H_{12}O_3S$. MW, 200. Cryst. from AcOEt. M.p. 33–4°. B.p. 173°/15 mm. D_4^{48} 1·166.
Propyl ester: $C_{10}H_{14}O_3S$. MW, 214. B.p. 154–6°/3 mm. D_4^{20} 1·144. n_D^{20} 1·4998.
Butyl ester: $C_{11}H_{16}O_3S$. MW, 228. B.p. 163–5°/3 mm. D_4^{20} 1·120. n_D^{20} 1·5050.
sec.-n-*Butyl ester*: decomp. on heating. D_4^{20} 1·140. n_D^{20} 1·5100.

Isobutyl ester : b.p. 163–5°/3 mm. D_4^{20} 1·125. n_D^{20} 1·5050.

n-*Amyl ester* : $C_{12}H_{18}O_3S$. MW, 242. B.p. 169–70°/3 mm. D_4^{20} 1·140. n_D^{20} 1·5100.

Phenyl ester : $C_{13}H_{12}O_3S$. MW, 248. Needles from EtOH. M.p. 95–6°. Very sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

Benzyl ester : $C_{14}H_{14}O_3S$. MW, 262. Cryst. M.p. 58°.

l-Menthyl ester : $C_{17}H_{26}O_3S$. MW, 310. Needles. M.p. 97°. $[\alpha]_D^{20}$ — 66·8° in $CHCl_3$. Spar. sol. cold EtOH.

1-*Naphthyl ester* : $C_{17}H_{14}O_3S$. MW, 298. Needles from EtOH. M.p. 83–4°.

2-*Naphthyl ester* : plates from EtOH or C_6H_6—ligroin. M.p. 125°.

Anhydride : $C_{14}H_{14}O_5S_2$. MW, 326. Cryst. from Et_2O. M.p. 122–5°.

Fluoride : toluene-*p*-sulphonfluoride. $C_7H_7O_2FS$. MW, 174. Needles from pet. ether. M.p. 43–4°. B.p. 112·5°/16 mm.

Chloride : toluene-*p*-sulphonchloride. $C_7H_7O_2ClS$. MW, 190·5. Cryst. from Et_2O or pet. ether. M.p. 71°. B.p. 145–6°/15 mm.

Bromide : toluene-*p*-sulphonbromide. $C_7H_7O_2BrS$. MW, 235. Columns. M.p. 96°.

Iodide : toluene-*p*-sulphoniodide. $C_7H_7O_2IS$. MW, 282. Yellow powder. M.p. 84–5° decomp.

Amide : *see* Toluene-*p*-sulphonamide.

Hydrazide : plates or needles from H_2O. M.p. 112°.

Phenylhydrazide : needles from EtOH. M.p. 155° decomp.

Azide : plates. M.p. 22°.

Anilide : needles from Et_2O–EtOH. M.p. 103°.

m-*Nitroanilide* : needles from EtOH.Aq. M.p. 139°.

p-*Nitroanilide* : yellow prisms from EtOH, needles from C_6H_6. M.p. 191°.

o-*Toluidide* : prisms from EtOH, needles from AcOH.Aq. M.p. 110°.

m-*Toluidide* : m.p. 114°.

p-*Toluidide* : needles from AcOH. M.p. 118–19°.

1-*Naphthylamide* : prisms from EtOH. M.p. 157°.

2-*Naphthylamide* : needles, plates or prisms from EtOH. M.p. 133°.

A,CH_3NH_2 : hygroscopic cryst. M.p. 125°.

A,$(CH_3)_2NH$: prisms. M.p. 78°.

A,$(CH_3)_3N$: rosettes. M.p. 92°.

A,$C_2H_5NH_2$: m.p. 111°.

A,$(C_2H_5)_2NH$: clusters of cryst. M.p. 88°.

A,$(C_2H_5)_3N$: m.p. 65°.

Curtius, Kraemer, *J. prakt. Chem.*, 1930, **125**, 323.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 22.

Gilman, Beaber, *J. Am. Chem. Soc.*, 1925, **47**, 522.

Holleman, Caland, *Ber.*, 1911, **44**, 2505.

Toluene-α-sulphonic Acid (*Benzylsulphonic acid*)

$C_6H_5{\cdot}CH_2{\cdot}SO_3H$

$C_7H_8O_3S$ MW, 172

Hygroscopic cryst. Forms spar. sol. Ba salt.

Phenyl ester : m.p. 80–1°.

Fluoride : $C_7H_7O_2FS$. MW, 174. Needles from petrol. M.p. 90–1°.

Chloride : $C_7H_7O_2ClS$. MW, 190·5. Prisms from Et_2O, needles from C_6H_6. M.p. 93°. Very sol. Et_2O, warm C_6H_6.

Amide : *see* Toluene-α-sulphonamide.

Hydrazide : plates from EtOH. M.p. 131–2° decomp.

Phenylhydrazide : m.p. 173° decomp.

Azide : needles from EtOH. M.p. 54°.

Anilide : needles from EtOH. M.p. 102°.

p-*Nitroanilide* : cryst. from EtOH.Aq. M.p. 155°.

o-*Toluidide* : m.p. 83°.

m-*Toluidide* : m.p. 75°.

p-*Toluidide* : prisms from EtOH. M.p. 113°.

1-*Naphthylamide* : yellow needles from EtOH. M.p. 166°.

2-*Naphthylamide* : needles. M.p. 148·5°.

Medwedew, Alexejewa, *Ber.*, 1932, **65**, 131.

Clutterbuck, Cohen, *J. Chem. Soc.*, 1923, **123**, 2507.

Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, **36**, 381.

Fromm, Palma, *Ber.*, 1906, **39**, 3312.

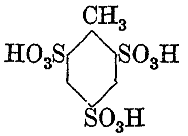
Toluenesulphoniodide.

See under Toluene-*p*-sulphonic Acid.

Toluene-tricarboxylic Acid.

See Methyltrimellitic Acid *and* Methyltrimesic Acid.

Toluene-2 : 4 : 6-trisulphonic Acid



$C_7H_8O_9S_3$ MW, 332

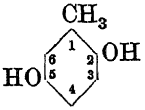
Needles + $6H_2O$ (after drying in vacuo). Retains $3H_2O$ at 100°. M.p. 145°. The trihydrate is hygroscopic. Very sol. H_2O.

Trichloride : $C_7H_5O_6Cl_3S_3$. MW, 387·5. Plates from $CHCl_3$. M.p. 153°. Spar. sol. boiling Et_2O.

Triamide : $C_7H_{11}O_6N_3S_3$. MW, 329. Cryst. Does not melt below 300°. Sol. warm NH_3. Insol. H_2O.

Claesson, *Ber.*, 1881, **14**, 307.

Toluhydroquinone (*Toluquinol, homohydroquinone, homoquinol, 2-methylhydroquinone, 2 : 5-dihydroxytoluene, hydrotoluquinone*)



$C_7H_8O_2$ MW, 124

Plates from C_6H_6. M.p. 124–5°. B.p. 163°/11 mm. Very sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6, ligroin. Sol. alkalis, alk. carbonates. Sublimes. Heat of comb. C_v 836·3 Cal. Reduces $NH_3.AgNO_3$ and Fehling's. $CrO_3 \longrightarrow$ *p*-toluquinone. $CaCl_2 \longrightarrow$ bluish-green $\longrightarrow$ brown col. $FeCl_3 \longrightarrow$ brownish-red or yellow col.

2-*Me ether*: $C_8H_{10}O_2$. MW, 138. Needles from C_6H_6–petrol. M.p. 46–46·5°. Very sol. cold EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. pet. ether, cold H_2O.

5-*Me ether*: needles from hot H_2O. M.p. 70·5–71·5°. B.p. 240–5°. Very sol. cold Et_2O, EtOH, $CHCl_3$. Spar. sol. pet. ether, cold H_2O.

Di-Me ether: $C_9H_{12}O_2$. MW, 152. Cryst. M.p. 15°. B.p. 214–18°. Volatile in steam.

5-*Et ether*: $C_9H_{12}O_2$. MW, 152. Prisms or needles from EtOH.Aq. M.p. 55–55·5°. Very sol. hot ligroin and most org. solvents. Spar. sol. hot H_2O. Volatile in steam.

Di-Et ether: $C_{11}H_{16}O_2$. MW, 180. Needles from cold ligroin. M.p. 24–5°. B.p. 247–9°.

Acetyl deriv.: needles from pet. ether. M.p. 92°.

Diacetyl: needles or prisms from H_2O, AcOH or ligroin. M.p. 49°.

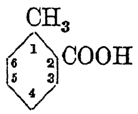
Dibenzoyl: cryst. M.p. 119–20°.

Schmid, *Monatsh.*, 1911, **32**, 437.
Bamberger, *Ann.*, 1912, **390**, 175.
Henderson, Boyd, *J. Chem. Soc.*, 1910, **97**, 1667.

α-Toluic Acid.

See Phenylacetic Acid.

o-Toluic Acid (2-*Methylbenzoic acid*)



$C_8H_8O_2$ MW, 136

Prisms from H_2O. M.p. 107–8° (102°). B.p. 258·5–259°. Very sol. EtOH. Sol. hot H_2O. Volatile in steam. Heat of comb. C_v 929·8 Cal., C_p 929·4 Cal. $k = 1{\cdot}35 \times 10^{-4}$ at 25°. Ox. $\longrightarrow$ phthalic acid. Na + amyl alcohol $\longrightarrow$ *trans.*-hexahydro-*o*-toluic acid.

Na salt: plates + $2H_2O$. M.p. 227–8°.

K salt: m.p. 188–9°.

Me ester: $C_9H_{10}O_2$. MW, 150. Cryst. below − 50°. B.p. 213°, 97°/15 mm. D^{15} 1·073.

Et ester: $C_{10}H_{12}O_2$. MW, 164. B.p. 227°, 102–102·5°/13 mm. $D_4^{21\cdot5}$ 1·0325. $n_D^{21\cdot6}$ 1·507.

d-*Amyl ester*: $C_{13}H_{18}O_2$. MW, 206. B.p. 265–8°. D_4^{20} 0·985. $n_D^{19\cdot6}$ 1·4984. $[\alpha]_D^{20}$ + 5·94°.

Allyl ester: b.p. 148°/45 mm.

l-*Menthyl ester*: $C_{18}H_{26}O_2$. MW, 274. B.p. 191°/15 mm. D_4^{20} 0·9982. $[\alpha]_D^{20}$ − 84·35°.

Phenyl ester: $C_{14}H_{12}O_2$. MW, 212. Yellow oil. B.p. 306°/754 mm.

Benzyl ester: $C_{15}H_{14}O_2$. MW, 226. Oil. B.p. 315°. D^{17} 1·12.

p-*Bromobenzyl ester*: m.p. 46°.

p-*Nitrobenzyl ester*: $C_{15}H_{13}O_4N$. MW, 271. Cryst. from EtOH.Aq. M.p. 90·7°.

p-*Bromphenacyl ester*: $C_{16}H_{13}O_3Br$. MW, 333. M.p. 56·9°.

Anhydride: $C_{16}H_{14}O_3$. MW, 254. Cryst. from Et_2O or C_6H_6. M.p. 39°. B.p. 220–1°/11 mm.

Chloride: C_8H_7OCl. MW, 154·5. B.p. 206–8°, 110–11°/29 mm., 99–100°/14 mm.

Bromide: C_8H_7OBr. MW, 199. B.p. 133–6°/37 mm.

Amide: *see* *o*-Toluamide.

Nitrile: *see* *o*-Tolunitrile.

Imide: cryst. from EtOH. M.p. 147–8°.

Hydrazide: needles from EtOH.Aq. or Et_2O. M.p. 124°.

Anilide: m.p. 125°.

p-*Toluidide*: m.p. 144°.

Clarke, Taylor, *Organic Syntheses*, 1931, XI, 96.
Zaugg, Rapala, *Organic Syntheses*, 1947, XXVII, 84.

m-Toluic Acid (3-*Methylbenzoic acid*).

Prisms from H_2O. M.p. 111–13°. B.p. 263°. Sol. 1170 parts H_2O at 15°, 60 parts at 100°. Very sol. EtOH, Et_2O. Sublimes. Volatile in steam. Heat of comb. C_v 928·5 Cal., C_p 929·1 Cal. $k = 5{\cdot}6 \times 10^{-5}$ at 25°. Ox. $\longrightarrow$ isophthalic acid. Red. $\longrightarrow$ hexahydro-*m*-toluic acid.

Na salt: m.p. about 310°.

Me ester: oil. B.p. 215°. D^{15} 1·066.

Et ester: b.p. 227°, 133°/38 mm., 103–5°/10 mm. D_{20}^{20} 1·0301. $n_D^{21\cdot6}$ 1·505.

Propyl ester: $C_{11}H_{14}O_2$. MW, 178. B.p. 240°.

d-*Amyl ester*: b.p. 266–8°/725 mm. D^{20} 0·976. n_D 1·4929. $[\alpha]_D^{20}$ + 6·59°.

l-*Menthyl ester*: b.p. 228–9°/36 mm., 197°/15 mm. D_4^{20} 0·9931. $[\alpha]_D^{20}$ − 87·59°.

Benzyl ester: b.p. 125–7°/15 mm. D_4^{21} 1·096. n_D^{20} 1·5652.

p-*Nitrobenzyl ester*: cryst. from EtOH.Aq. M.p. 86·6°.

p-*Bromphenacyl ester*: m.p. 108°.

Anhydride: cryst. from pet. ether. M.p. 71°. B.p. 230°/17 mm. Sol. most org. solvents.

Chloride: m.p. − 23°. B.p. 219–20°/773 mm., 136–8°/31 mm., 109°/15 mm. D_4^{20} 1·173.

Bromide: b.p. 136–7°/52 mm.

Amide: *see* *m*-Toluamide.

Nitrile: *see* *m*-Tolunitrile.

Hydrazide: plates from EtOH.Aq. M.p. 97°.

Adams, Ulich, *J. Am. Chem. Soc.*, 1920, **42**, 608.
Reuter, *Ber.*, 1884, **17**, 2028.
Emerson, Lucas, Heimsch, *J. Am. Chem. Soc.*, 1949, **71**, 1742.

p-Toluic Acid (4-*Methylbenzoic acid*).

Cryst. from hot H_2O. M.p. 181°. B.p. 274–5°. Very sol. EtOH, MeOH, Et_2O. Spar. sol. H_2O. Volatile in steam. Sublimes. Heat of comb. C_p 927·4 Cal., C_v 926·8 Cal. $k = 4{\cdot}3 \times 10^{-5}$ at

25°. Dist. with CaO ⟶ toluene. Ox. ⟶ terephthalic acid. Na + amyl alcohol ⟶ hexahydro-*p*-toluic acid.

Me ester: cryst. with intense unpleasant odour from MeOH.Aq. or pet. ether. M.p. 33·2°. B.p. 217°.

Et ester: b.p. 228°, 122°/22 mm., 110°/12 mm. D_{25}^{25} 1·024. $n_D^{18\cdot2}$ 1·5089.

d-*Amyl ester*: b.p. 271–2°. D_4^{20} 0·982. $n_D^{19\cdot1}$ 1·4975. $[\alpha]_D^{20}$ + 6·67°.

Isoamyl ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 271°.

l-*Menthyl ester*: cryst. M.p. 40–1°. B.p. 196–8°/11 mm. $[\alpha]_D^{20}$ − 89·93°.

Phenyl ester: plates from EtOH. M.p. 83°.

Benzyl ester: m.p. 45–7°. B.p. 124–6°/ 0·25–0·5 mm.

p-*Bromobenzyl ester*: m.p. 72°.

p-*Nitrobenzyl ester*: cryst. from EtOH.Aq. M.p. 104·5°.

p-*Bromphenacyl ester*: m.p. 153°.

Anhydride: plates from MeOH, needles from EtOH. M.p. 95°. Stable to boiling H_2O.

Chloride: m.p. − 2 to − 1·5°. B.p. 214–16°, 102°/15 mm.

Bromide: b.p. 145–9°/42 mm.

Amide: *see* *p*-Toluamide.

Nitrile: *see* *p*-Tolunitrile.

Imide: needles from C_6H_6. M.p. 155°.

Hydrazide: plates from EtOH.Aq. M.p. 117°.

Anilide: needles or plates from EtOH. M.p. 147–8°.

o-*Nitroanilide*: yellow prisms from EtOH. M.p. 110°.

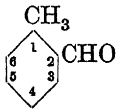
p-*Toluidide*: needles from EtOH. M.p. 165° (158–9°).

Herb, *Ann.*, 1890, **258**, 10.
Gattermann, Schmidt, *Ann.*, 1888, **244**, 51.
Tuley, Marvel, *Organic Syntheses*, 1947, XXVII, 86.

α-Toluic Aldehyde.

See Phenylacetaldehyde.

***o*-Toluic Aldehyde** (2-*Methylbenzaldehyde*)



C_8H_8O MW, 120

B.p. 197°, 94°/10 mm. D_4^{19} 1·0386. n_D^{19} 1·549. KCN + EtOH ⟶ *o*-toluoin. Ox. in air ⟶ *o*-toluic acid. Red. ⟶ *o*-tolylcarbinol. Sol. H_2SO_4 ⟶ greenish-orange sol.

Oxime: needles from pet. ether. M.p. 49°. Very sol. hot H_2O, EtOH, Et_2O, CS_2, C_6H_6. Spar. sol. pet. ether. N-*Acetyl*: cryst. from Et_2O. M.p. 55–6°.

Semicarbazone: needles from amyl alcohol. M.p. 212° (196°).

Hydrazone: m.p. 97°.

Phenylhydrazone: m.p. 105–6°.

p-*Nitrophenylhydrazone*: red needles from EtOH. M.p. 222°.

2 : 4-*Dinitrophenylhydrazone*: orange-red plates from AcOH. M.p. 193–4°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, **13**, 460.
Stephen, *J. Chem. Soc.*, 1925, 1874.
Gattermann, *Ann.*, 1912, **393**, 218.
Gattermann, Maffezoli, *Ber.*, 1903, **36**, 4152.
Williams, Witten, Krynitsky, *Organic Syntheses*, 1946, XXVI, 97.

***m*-Toluic Aldehyde** (3-*Methylbenzaldehyde*).

B.p. 199°, 93–4°/17 mm. $D_4^{21\cdot4}$ 1·0189. $n_D^{21\cdot4}$ 1·541. Oxidises in air to *m*-toluic acid. HNO_3 ⟶ isophthalic acid.

Semicarbazone: needles from EtOH, plates from amyl alcohol. M.p. 223–4° (206°).

Phenylhydrazone: prisms from ligroin or EtOH.Aq. M.p. 90°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, **13**, 462.
Sommelet, *Compt. rend.*, 1913, **157**, 853.
Smith, Bayliss, *J. Org. Chem.*, 1941, **6**, 437.

***p*-Toluic Aldehyde** (4-*Methylbenzaldehyde*).

Liq. with peppermint odour. B.p. 204–5°, 106°/10 mm. $D_4^{16\cdot7}$ 1·0194. $n_D^{16\cdot6}$ 1·547. KCN+ EtOH ⟶ 4 : 4′-dimethylbenzoin. Oxidises in air to *p*-toluic acid. Forms cryst. bisulphite comp. Sol. H_2SO_4 ⟶ orange-brown sol.

Polymer: $(C_8H_8O)_x$. Cryst. from C_6H_6. M.p. 215°. Insol. cold EtOH, Et_2O, AcOH.

Di-Et acetal: $C_{12}H_{18}O_2$. MW, 194. B.p. 116–19°/12 mm. D^{22} 1·006. n^{22} 1·47603.

NH_3 add. comp.: cryst. M.p. 43–4°.

Oxime: *syn*-. M.p. 108–10°. Steam dist. of alk. sol. ⟶ *anti*-. N-*Acetyl*: m.p. 85°. *Anti*-: m.p. 79–80°.

Semicarbazone: needles from EtOH, plates from amyl alcohol. M.p. 234° (215°).

Hydrazone: cryst. mass. M.p. 56°. B.p. 148°/12 mm.

Phenylhydrazone: plates from EtOH. M.p. 121° (108°).

o-*Nitrophenylhydrazone*: red needles from EtOH.Aq. M.p. 183°.

m-*Nitrophenylhydrazone*: m.p. 155°.

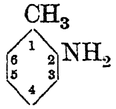
p-*Nitrophenylhydrazone*: dark red needles with green fluor. from EtOH. M.p. 200·5°.

2 : 4-*Dinitrophenylhydrazone*: orange-yellow cryst. from $PhNO_2$. M.p. 232·5–234·5°.

p-*Bromphenylhydrazone*: m.p. 162° decomp.

I.C.I., B.P. 397,124, (*Chem. Abstracts*, 1934, **28**, 778).
Coleman, Craig, *Organic Syntheses*, 1932, XII, 80.
Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2793.
Gattermann, Koch, *Ber.*, 1897, **30**, 1623.
Smith, Nichols, *J. Org. Chem.*, 1941, **6**, 489.

o-Toluidine (*2-Aminotoluene, o-tolylamine*)



C_7H_9N MW, 107

Liq. Cryst. on cooling to two forms. α-. M.p. about − 21°. β- (stable form). M.p. about − 15·5°. B.p. 200·6°/754·6 mm., 121°/80 mm. D_{20}^{20} 1·0053. n_D^{20} 1·5688. Sol. EtOH, Et_2O. Spar. sol. H_2O. Specific heat 0·49 cal./gm. at 15–64°. Heat of comb. C_v 963·8 Cal., C_p 969·93 Cal. $k = 3{\cdot}5 \times 10^{-10}$ at 25°. Volatile in steam. $MnO_2 + H_2SO_4 \longrightarrow$ toluquinone. $KMnO_4 \longrightarrow$ 2 : 2′-dimethylazobenzene. Dil. H_2SO_4 sol. + CrO_3 in dil. $H_2SO_4 \longrightarrow$ blue col. $\longrightarrow$ reddish-violet col. on dilution. Forms Ca and Na derivs.

$C_7H_9N,C_6H_3(NO_2)_3$-1 : 3 : 5 : light red needles. M.p. 125–7°.

B,HCl : cryst. M.p. 215°. B.p. 242·2°.

B,H_3PO_3 : needles. M.p. 174°.

$B,HClO_3$: plates from EtOH. Explodes at 88°.

B_2,H_2PtBr_6 : yellowish-red needles. M.p. 225–6° decomp.

$B,CH_2(COOH)_2$: prisms. Decomp. at 108°.

$B,ClCH_2{\cdot}COOH$: m.p. 95°.

$B_2,(COOH)_2$: m.p. 167°.

$B,(COOH)_2$: plates. M.p. 171°.

$B,(CH_2{\cdot}COOH)_2$: prisms. Decomp. at 60°.

$B_2,ZnCl_2$: cryst. from EtOH. M.p. 227°.

$B_2,ZnBr_2$: cryst. from EtOH. M.p. 218°.

N-*Me* : *see* *N*-Methyl-*o*-toluidine.

N-*Di-Me* : *see* *N*-Dimethyl-*o*-toluidine.

N-*Et* : *see* *N*-Ethyl-*o*-toluidine.

N-*Di-Et* : *see* Diethyl-*o*-toluidine.

N-*Propyl* : *see* *N*-Propyl-*o*-toluidine.

N-*Butyl* : $C_{11}H_{17}N$. MW, 163. Oil with pleasant odour. B.p. 258–60°/771 mm.

N-*Isobutyl* : *see* *N*-Isobutyl-*o*-toluidine.

N-*Isoamyl* : *see* *N*-Isoamyl-*o*-toluidine.

N-*Allyl* : $C_{10}H_{13}N$. MW, 147. B.p. 225–30°.

N-β-*Hydroxyethyl* : *see* *N*-β-Hydroxyethyl-*o*-toluidine.

N-*Phenyl* : *see* 2-Methyldiphenylamine.

N-*Tolyl* : *see* Ditolylamine.

N-*Benzyl* : *see* *N*-Benzyl-*o*-toluidine.

N-*Dibenzyl* : $C_{21}H_{21}N$. MW, 287. Cryst. M.p. 42°. B.p. 223°/10 mm. D_4^{65} 1·02347. n_D^{65} 1·58324.

N-*Picryl* : $C_{13}H_{10}O_6N_4$. MW, 318. Orange-red prisms from EtOH–Me_2CO. M.p. 164°.

N-*Formyl* : *see* Formo-*o*-toluidide.

N-*Acetyl* : *see* Acet-*o*-toluidide.

N-*Diacetyl* : *N*-*o*-tolyldiacetamide. Cryst. M.p. 18°. B.p. 200·5–201°/100 mm., 144–5°/11 mm.

N-*Benzoyl* : benz-*o*-toluidide. Needles from AcOEt–Me_2CO. M.p. 145–6°.

N-*Dibenzoyl* : *N*-*o*-tolyldibenzamide. Prisms from EtOH. M.p. 111–12°.

N-p-*Toluenesulphonyl* : m.p. 185·5–186·2°.

N-1-*Naphthalenesulphonyl* : m.p. 237°.

Picrate : cryst. from EtOH. M.p. 212–15° decomp.

Tanner, Lasselle, *J. Am. Chem. Soc.*, 1926, **48**, 2163.
Courtot, Petitcolas, *Bull. soc. chim.*, 1926, **39**, 452.
Blanksma, *Rec. trav. chim.*, 1909, **28**, 109.

m-Toluidine (*3-Aminotoluene, m-tolylamine*). Liq. Cryst. on strong cooling. M.p. − 43·6°. B.p. 203·2°. D_{25}^{25} 0·990. n_D^{20} 1·56859. Sol. EtOH, Et_2O. Very spar. sol. H_2O. Heat of comb. C_v 964·6 Cal., C_p 965·6 Cal. $k = 5{\cdot}5 \times 10^{-10}$ at 25°. Volatile in steam. Ox. $\longrightarrow$ toluquinone. Sol. in dil. $H_2SO_4 + CrO_3$ in dil. $H_2SO_4 \longrightarrow$ yellowish-brown col. Sol. in dil. $H_2SO_4 + HNO_3 \longrightarrow$ dark red col.

$C_7H_9N,C_6H_3(NO_2)_3$-1 : 3 : 5 : light red needles. M.p. 93°.

B,HCl : plates from H_2O. M.p. 228°. B.p. 249·8°.

$B,HClO_4$: m.p. 200° decomp.

B_2,H_2PtBr_6 : red scales. M.p. 266° decomp.

$B,CH_2(COOH)_2$: prisms. Decomp. at 93°.

$B,(CH_2{\cdot}COOH)_2$: columns. Decomp. at 121°.

N-*Me* : *see* *N*-Methyl-*m*-toluidine.

N-*Di-Me* : *see* Dimethyl-*m*-toluidine.

N-*Et* : *see* *N*-Ethyl-*m*-toluidine.

N-*Di-Et* : *see* Diethyl-*m*-toluidine.

N-*Phenyl* : *see* 3-Methyldiphenylamine.

N-*Diphenyl* : *see* Diphenyl-*m*-toluidine.

N-*Tolyl* : *see* Ditolylamine.

N-*Benzyl* : *see* *N*-Benzyl-*m*-toluidine.

N-*Dibenzyl* : $C_{21}H_{21}N$. MW, 287. Cryst. M.p. 78°. B.p. 229°/10 mm.

N-*Picryl* : two forms. α-. Yellow prisms from EtOH–HCl. M.p. 130°. β-. Orange-red needles from alc. NH_3. M.p. 129·5°.

N-*Formyl* : *see* Formo-*m*-toluidide.

N-*Acetyl* : *see* Acet-*m*-toluidide.

N-*Benzoyl* : benz-*m*-toluidide. Prisms from EtOH.Aq. M.p. 125°.

N-*Dibenzoyl* : *N*-*m*-tolyldibenzamide. Needles from EtOH. M.p. 140–1°.

N-p-*Toluenesulphonyl* : m.p. 171–172·5°.

N-1-*Naphthalenesulphonyl* : m.p. 195–6°.

Buchka, Schachtebeck, *Ber.*, 1889, **22**, 840.
Blanksma, *Rec. trav. chim.*, 1906, **25**, 370.
Ehrlich, *Ber.*, 1882, **15**, 2011.

p-Toluidine (*4-Aminotoluene, p-tolylamine*). Cryst. + $1H_2O$ from H_2O or EtOH.Aq. M.p. 42°, anhyd. 44·5–45°. Anhyd. in moist air $\longrightarrow$ monohydrate. B.p. 200·3°, 133·7°/100 mm., 100·2°/25 mm., 82·2°/10 mm. Sol. 285 parts H_2O at 11·5°. Very sol. EtOH.Aq., MeOH, Et_2O, Me_2CO, CS_2. Volatile in steam. D_{50}^{50}

0·973. $n_D^{59\cdot1}$ 1·55324. Heat of comb. C_v 957·9 Cal., C_p 958·8 Cal., C_p (liq.) 973·5 Cal. $k = 1{\cdot}48 \times 10^{-9}$ at 25°. Alk. $KMnO_4 \longrightarrow$ 4 : 4′-dimethylazobenzene. Acid $KMnO_4 \longrightarrow$ *p*-nitrosotoluene. Reduces alc. $NH_3.AgNO_3$ in the cold. Dil. H_2SO_4 sol. + $HNO_3 \longrightarrow$ blue $\longrightarrow$ violet $\longrightarrow$ red $\longrightarrow$ brown col. Slightly acid sol. + $FeCl_3 \longrightarrow$ pale yellow $\longrightarrow$ red col. Forms metallic derivs. with Ca and Na.

B,HCl: needles from AcOH–Et_2O. M.p. 243°. B.p. 257·5°.

B_2,H_2PtBr_6: yellowish-red cryst. M.p. 268–9°.

B,$ClCH_2$·COOH: needles. M.p. 101–2°.

B,Cl_2CH·COOH: needles. M.p. 140–1°.

B,Cl_3C·COOH: cryst. M.p. 137°.

B_2,$(COOH)_2$: m.p. 183–4°.

B,$(COOH)_2$: m.p. 178°.

B,$CH_2(COOH)_2$: m.p. 114°.

B,$(CH_2$·$COOH)_2$: m.p. 123–4°.

B,C_6H_5·COOH: m.p. 52·5°.

B_2,$AgNO_3$: plates. M.p. 101° decomp.

N-*Me*: *see* *N*-Methyl-*p*-toluidine.

N-*Di-Me*: *see* Dimethyl-*p*-toluidine.

N-*Et*: *see* *N*-Ethyl-*p*-toluidine.

N-*Di-Et*: *see* Diethyl-*p*-toluidine.

N-*Propyl*: *see* *N*-Propyl-*p*-toluidine.

N-*Isopropyl*: *see* *N*-Isopropyl-*p*-toluidine.

N-*Butyl*: $C_{11}H_{17}N$. MW, 163. Oil. B.p. 264–5°/766 mm. Volatile in steam. *B,HCl*: needles or prisms from EtOH. M.p. 150–1°.

N-*Dibutyl*: $C_{15}H_{25}N$. MW, 219. Oil. B.p. 282–4°/764 mm. *Picrate*: yellow cryst. from Et_2O–pet. ether. M.p. 131–2°.

N-*Isobutyl*: *see* *N*-Isobutyl-*p*-toluidine.

N-*Allyl*: $C_{10}H_{13}N$. MW, 147. Oil. B.p. 232–4°. *B,HCl*: m.p. 131–2°.

N-β-*Hydroxyethyl*: *see* *N*-β-Hydroxyethyl-*p*-toluidine.

N-*Phenyl*: *see* 4-Methyldiphenylamine.

N-*Tolyl*: *see* Ditolylamine.

N-*Benzyl*: *see* *N*-Benzyl-*p*-toluidine.

N-*Dibenzyl*: $C_{21}H_{21}N$. MW, 287. Cryst. M.p. 56°. B.p. 233°/11 mm. D_4^{65} 1·03721. n_D^{65} 1·60109.

N-*Picryl*: two forms. α-. Yellow needles from Me_2CO, $CHCl_3$, CCl_4 or C_6H_6. M.p. 164°. β-. Dark red needles from Py. M.p. 164°.

N-*Bornyl*: $C_{17}H_{25}N$. MW, 243. Needles. M.p. 33°. B.p. 162°/3 mm. Very sol. EtOH, Et_2O, C_6H_6. *B,HCl*: cryst. powder. M.p. 214° decomp.

N-*Formyl*: *see* Formo-*p*-toluidide.

N-*Acetyl*: *see* Acet-*p*-toluidide.

N-*Diacetyl*: *N*-*p*-tolyldiacetamide. Cryst. M.p. 48°. B.p. 160–1°/15 mm.

N-*Benzoyl*: benz-*p*-toluidide. Needles from EtOH. M.p. 158°. B.p. 232°. N-*Nitroso*: needles from Me_2CO.Aq. M.p. 75° decomp.

N-*Dibenzoyl*: *N*-*p*-tolyldibenzamide. Prisms from EtOH. M.p. 142–4°.

N-p-*Toluenesulphonyl*: m.p. 193–194·5°.

N-1-*Naphthalenesulphonyl*: m.p. 181°.

Picrate: m.p. 180–1° decomp.

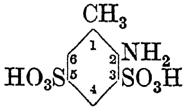
Blanksma, *Rec. trav. chim.*, 1909, **28**, 109; 1906, **25**, 370.

Popov, *Chem. Abstracts*, 1934, **28**, 1671.

Kock, *Ber.*, 1887, **20**, 1568.

Graebe, *Ber.*, 1901, **34**, 1778.

o-Toluidine-3 : 5-disulphonic Acid



$C_7H_9O_6NS_2$ MW, 267

Needles + $1\frac{1}{2}H_2O$ from H_2O. Decomp. at 240°. Very sol. H_2O, EtOH. Insol. C_6H_6.

Dichloride: $C_7H_7O_4NCl_2S_2$. MW, 304. Cryst. from C_6H_6–pet. ether. M.p. 153°.

Dianilide: cryst. from EtOH.Aq. M.p. 188°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 128.

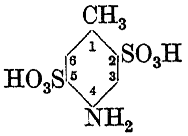
Nevile, Winther, *Ber.*, 1882, **15**, 2992.

o-Toluidine-4 : 5-disulphonic Acid.

Needles. Spar. sol. Forms spar. sol. Ba salt.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 745.

p-Toluidine-2 : 5-disulphonic Acid



$C_7H_9O_6NS_2$ MW, 267

Needles + $2\frac{1}{2}H_2O$. Decomp. at 290°. Very sol. H_2O. Spar. sol. EtOH. Forms spar. sol. Ba salt.

Dichloride: $C_7H_7O_4NCl_2S_2$. MW, 304. Yellow prisms from $CHCl_3$. M.p. 156°. Sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. N-*Acetyl*: cryst. from pet. ether. M.p. 125°. N-*Chloracetyl*: cryst. from pet. ether. M.p. 118–19°.

Diamide: $C_7H_{11}O_4N_3S_2$. MW, 265. Needles from H_2O. M.p. 257°. Sol. H_2O, EtOH, Me_2CO.

Dianilide: plates from EtOH.Aq. M.p. 196–7°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 126.

Riesz, Pollak, Wittels, *Ann.*, 1931, **487**, 267.

Lustig, Katscher, *Monatsh.*, 1927, **48**, 93.

p-Toluidine-2 : 6-disulphonic Acid.

Prisms + H_2O. Very sol. H_2O. Spar. sol. EtOH. Ppd. by EtOH from H_2O.

Kornatzki, *Ann.*, 1883, **221**, 198.

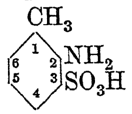
p-Toluidine-3 : 5-disulphonic Acid.

Needles + $2H_2O$, aggregates of cryst. + $1H_2O$. Anhyd. at 120°. Decomp. at 200°. Very sol. H_2O. Mod. sol. EtOH. Decomp. with H_2O at 140° $\longrightarrow$ *p*-toluidine-3-sulphonic acid.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 734.

Richter, *Ann.*, 1885, **230**, 315.

o-Toluidine-3-sulphonic Acid



$C_7H_9O_3NS$ MW, 187

Needles. Very sol. hot H_2O. Warm with aq. $FeCl_3$ ⟶ reddish-yellow col.

Pechmann, *Ann.*, 1874, **173**, 215.

o-Toluidine-4-sulphonic Acid.

Needles or prisms + $1H_2O$. Anhyd. over H_2SO_4. Sol. 104 parts H_2O at 11°. Insol. EtOH. $k = 2{\cdot}5 \times 10^{-4}$ at 25°. $FeCl_3$ ⟶ dark violet col. NaOH fusion ⟶ anthranilic acid.

Fluoride: $C_7H_8O_2NFS$. MW, 189. Needles from EtOH.Aq. M.p. 96–7°. Very sol. Me_2CO. Sol. Et_2O, AcOEt, C_6H_6. Mod. sol. EtOH, CS_2, pet. ether. Spar. sol. H_2O. N-*Acetyl*: needles from EtOH. M.p. 188·5–189·5°.

Chloride: $C_7H_8O_2NClS$. MW, 205·5. N-*Acetyl*: prisms from C_6H_6. M.p. 144°. N-*Benzoyl*: needles from C_6H_6. M.p. 196°.

Amide: $C_7H_{10}O_2N_2S$. MW, 186. Columns. M.p. 176°. Very sol. hot H_2O. Spar. sol. EtOH. Insol. Et_2O, C_6H_6. *B,HCl*: needles. M.p. 240°.

N-*Me*: $C_8H_{11}O_3NS$. MW, 201. Plates from hot H_2O. Sol. 60 parts H_2O at 15°.

N-*Di-Et*: $C_{11}H_{17}O_3NS$. MW, 243. Plates + $1H_2O$ from H_2O. Spar. sol. cold H_2O, EtOH.

N-*Benzoyl*: plates. M.p. 203°.

I.G., D.R.P., 573,193, (*Chem. Zentr.*, 1933, II, 445).

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 26.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 745.

o-Toluidine-5-sulphonic Acid.

Plates or columns from $1H_2O + H_2O$. Anhyd. at 120° over H_2SO_4. Sol. 32 parts H_2O at 19°. $k = 7{\cdot}53 \times 10^{-4}$ at 25°. Characteristic col. with PbO_2 in aq. sol.

N-*Me*: needles from H_2O. Sol. H_2O.

N-*Di-Me*: $C_9H_{13}O_3NS$. MW, 215. Prisms from H_2O. Very sol. hot H_2O. Insol. EtOH.

Schultz, Lucas, *J. Am. Chem. Soc.*, 1927, **49**, 299.

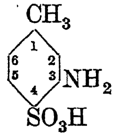
Nevile, Winther, *Ber.*, 1880, **13**, 1941.

o-Toluidine-6-sulphonic Acid.

Needles. Sol. 293 parts H_2O at 22°.

Pagel, *Ann.*, 1875, **176**, 305.

m-Toluidine-4-sulphonic Acid



$C_7H_9O_3NS$ MW, 187

Needles + H_2O. Sol. 715 parts H_2O at 16°.

Hayduck, *Ann.*, 1874, **174**, 350.

m-Toluidine-6-sulphonic Acid.

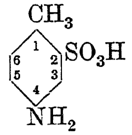
Plates. Chars above 275°. Spar. sol. H_2O. $k = 3{\cdot}57 \times 10^{-4}$ at 25°.

NH_4 salt: m.p. 190°.

Quilico, *Gazz. chim. ital.*, 1926, **56**, 626.

Seyewetz, Bloch, *Bull. soc. chim.*, 1907, **1**, 327.

p-Toluidine-2-sulphonic Acid



$C_7H_9O_3NS$ MW, 187

Prisms + $1H_2O$. Decomp. at high temps. Sol. 220 parts H_2O at 20°. Insol. EtOH. $k = 4{\cdot}08 \times 10^{-5}$ at 25°. $FeCl_3$ ⟶ red col. Reduces $NH_3.AgNO_3$ on warming.

Fluoride: $C_7H_8O_2NFS$. MW, 189. Yellow cryst. from EtOH. M.p. 62°. Sol. Et_2O, AcOEt, C_6H_6. Insol. H_2O. N-*Acetyl*: needles from C_6H_6. M.p. 120–1°.

Chloride: $C_7H_8O_2NClS$. MW, 205·5. N-*Acetyl*: prisms from Me_2CO.Aq. M.p. 124°. N-*Chloracetyl*: plates from petrol. M.p. 87°.

Amide: $C_7H_{10}O_2N_2S$. MW, 186. Needles or plates. M.p. 164°. Sol. hot H_2O, EtOH. N-*Acetyl*: cryst. from H_2O. M.p. 242°. N-*Chloracetyl*: needles from H_2O. M.p. 231°.

N-*Me*: $C_8H_{11}O_3NS$. MW, 201. Plates. Sol. H_2O. Insol. org. solvents. Decomp. on boiling aq. sol.

N-*Et*: $C_9H_{13}O_3NS$. MW, 215. Yellow prisms + $1H_2O$ from H_2O. Spar. sol. H_2O. Very spar. sol. EtOH.

N-*Di-Et*: $C_{11}H_{17}O_3NS$. MW, 243. Prisms + $1H_2O$ from H_2O. Does not melt. Spar. sol. H_2O. Insol. Et_2O.

N-*Isopropyl*: $C_{10}H_{15}O_3NS$. MW, 229. Prisms from H_2O. Does not melt below 300°. Spar. sol. EtOH.

N-*Acetyl*: needles + $2H_2O$ from dil. HCl. *Anilide*: leaflets from EtOH. M.p. 230°.

Anilide: m.p. 146–7°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 125.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 37.

Johnson, Smiles, *J. Chem. Soc.*, 1923, **123**, 2385.

p-Toluidine-3-sulphonic Acid.

Needles + $\frac{1}{2}H_2O$. Sol. 214 parts H_2O. $k = 8{\cdot}5 \times 10^{-4}$ at 25°. Aq. sol. + PbO_2 ⟶ red col. Decomp. with H_2O at 130°. Gives Ag salt, spar. sol. H_2O at 100°.

N-*Me*: $C_8H_{11}O_3NS$. MW, 201. Prisms from H_2O. Decomp. on heating.

N-*Di-Et*: prisms + $1H_2O$ from H_2O. M.p. 243°. Sol. H_2O. Insol. Et_2O, C_6H_6. *K salt*: plates + $1\frac{1}{2}H_2O$. M.p. 297°.

N-*Di-isopropyl*: $C_{13}H_{21}O_3NS$. MW, 271. Cryst. + $2H_2O$ from H_2O. M.p. 222–3°.

Leitch, B.P. 257,979, (*Chem. Zentr.*, 1927, I, 1745).
Witt, Uerményi, *Ber.*, 1913, **46**, 301.
Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 738.

Toluidinoacetic Acid.
See Tolylglycine.

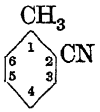
Toluidinobenzoic Acid.
See Methyldiphenylamine-carboxylic Acid.

2-Toluidinoethyl Alcohol.
See *N*-β-Hydroxyethyltoluidine.

Toluidinophenol.
See Hydroxy-methyldiphenylamine.

Tolunitranilic Acid.
See 5-Nitro-3 : 6-dihydroxytoluquinone.

***o*-Tolunitrile** (o-*Cyanotoluene*)



C_8H_7N MW, 117

Liq. M.p. − 13 to − 14°. B.p. 205·2°, 90°/15 mm., 82·3°/11 mm. D_{25}^{25} 0·9912. n_D^{25} 1·52720. Heat of comb. C_v 1030 Cal., C_p 1030·7 Cal. Red. ⟶ *o*-xylylamine. Hyd. ⟶ *o*-toluic acid.

Clarke, Read, *Organic Syntheses*, Collective Vol. I, 500.

***m*-Tolunitrile** (m-*Cyanotoluene*).

Liq. M.p. − 23·5 to − 23°. B.p. 210–12°, 84·5°/10 mm. D^{20} 1·0316. Red. ⟶ *m*-xylylamine. H_2O_2 ⟶ *m*-toluamide.

Bayer, D.R.Ps., 259,363, 259,364, (*Chem. Zentr.*, 1913, I, 1741).
Buchka, Schachtebeck, *Ber.*, 1889, **22**, 841.

***p*-Tolunitrile** (p-*Cyanotoluene*).

Needles from EtOH. M.p. 29°. B.p. 217·6°, 90·5–91°/11 mm. D_{30}^{30} 0·9805. $KMnO_4$ ⟶ terephthalamic acid. H_2O_2 ⟶ *p*-toluamide.

Clarke, Read, *Organic Syntheses*, Collective Vol. I, 500.

Toluoin.
See Dimethylbenzoin.

Toluylenediamine.
See Tolylenediamine.

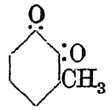
Toluquinaldine.
See 2 : 6-, 2 : 7-, *and* 2 : 8-Dimethylquinoline.

Toluquinhydrone.
See under Toluquinone.

Toluquinol.
See Toluhydroquinone.

Toluquinoline.
See 6-, 7-, *and* 8-Methylquinoline.

***o*-Toluquinone** (3-*Methyl*-o-*benzoquinone*)



$C_7H_6O_2$ MW, 122

Dark red prisms and needles from Et_2O or pet. ether. Sol. Et_2O ⟶ green sol. Warm, or standing in Et_2O ⟶ dimer.

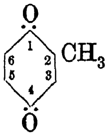
Dimer: $C_{14}H_{12}O_4$. MW, 244. Yellow prisms and plates from $CHCl_3$–pet. ether. M.p. 194–5°. Very sol. $CHCl_3$. Insol. Et_2O, pet. ether. Gives no quinone reactions.

1-*Oxime*: *see* 3-Nitroso-*o*-cresol.

Dioxime: yellowish-brown needles from H_2O. M.p. about 140° decomp. Sol. EtOH, AcOH. Mod. sol. H_2O, C_6H_6.

Willstätter, Müller, *Ber.*, 1911, **44**, 2178.

Toluquinone (p-*Toluquinone*, *methylquinone*, 2-*methyl*-p-*benzoquinone*)



$C_7H_6O_2$ MW, 122

Yellow plates or needles. M.p. 69°. Sol. EtOH, Et_2O. Spar. sol. cold H_2O. Volatile in steam. Sublimes. Heat of comb. C_v 805·0 Cal., C_p 805·3 Cal. Mod. conc. H_2SO_4 ⟶ polymer. Red. ⟶ toluhydroquinone. Triphenylmagnesium chloride ⟶ toluquinhydrone. Aq. sol. + alkali ⟶ brownish-red col.

Polymer: powder. Does not melt below 300°. Very sol. EtOH, Et_2O. Sol. AcOH. Spar. sol. H_2O. Insol. C_6H_6.

Toluhydroquinone comp.: toluquinhydrone. $C_{14}H_{14}O_4$. MW, 246. Black needles. M.p. 96–7°.

1-*Oxime*: *see* 6-Nitroso-*m*-cresol.

4-*Oxime*: *see* 5-Nitroso-*o*-cresol.

1-*Oxime*-4-*imide*: *see* 6-Nitroso-*m*-toluidine.

4-*Oxime*-1-*imide*: *see* 5-Nitroso-*o*-toluidine.

Dioxime: needles. Decomp. at 234°. Sol. EtOH, Et_2O, hot H_2O. Spar. sol. $CHCl_3$, C_6H_6. Insol. ligroin. N-*Diacetyl*: needles from EtOH.Aq. M.p. 120°. N-*Benzoyl*: yellow cryst. from EtOH. M.p. 180° decomp.

1-*Chloroimide*: C_7H_6ONCl. MW, 155·5. Yellow columns from EtOH. M.p. 75°.

4-*Chloroimide*: yellow needles. M.p. 87–8°.

Dichloroimide: $C_7H_6N_2Cl_2$. MW, 189. Yellow needles. M.p. 74°. Spar. sol. H_2O. Sol. EtOH.

1-*Imide*-4-*semicarbazone*: monohydrated reddish-brown needles from Me_2CO–C_6H_6. M.p. 85–6° decomp.

4-*Semicarbazone*: yellow needles from EtOH. M.p. 178–9°.

1 : 4-*Disemicarbazone*: orange-red. M.p. 240° decomp.

4-Phenylsemicarbazone: dark red cryst. from EtOH. M.p. 198–9° decomp.

1 : *4-Diphenylsemicarbazone*: red. cryst. powder. Decomp. at 246°.

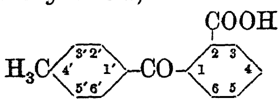
Di-2 : 4-dinitrophenylhydrazone: greyish-black needles from $PhNO_2$. M.p. 269°.

4-Benzoylphenylhydrazone: yellow plates from C_6H_6. M.p. 151°.

Clark, *Am. Chem. J.*, 1892, **14**, 565.
Borsche, Müller, Bodenstein, *Ann.*, 1929, **472**, 214.

Toluquinoxaline.
See 6-Methylquinoxaline.

2-*p*-Toluylbenzoic Acid (*4′-Methylbenzophenone-2-carboxylic acid*)



$C_{15}H_{12}O_3$ MW, 240

Prisms + $1H_2O$ from EtOH–toluene. M.p. 146° (139–40°). Sol. EtOH, Et_2O, Me_2CO, C_6H_6, boiling toluene. Spar. sol. H_2O. Ox. ⟶ benzophenone-2 : 4′-dicarboxylic acid.

Me ester: $C_{16}H_{14}O_3$. MW, 254. Plates from MeOH. M.p. 61° (53°). Sol. EtOH, C_6H_6. Pale yellow sol. in conc. H_2SO_4.

Et ester: $C_{17}H_{16}O_3$. MW, 268. Plates. M.p. 68–9°.

Amide: $C_{15}H_{13}O_2N$. MW, 239. Needles from H_2O. M.p. 175–6°. Sol. MeOH, Et_2O, AcOH. Spar. sol. C_6H_6, ligroin.

Friedel, Crafts, *Bull. soc. chim.*, 1881, **35**, 505.
Heller, Schülke, *Ber.*, 1908, **41**, 3632.
Fieser, *Organic Syntheses*, Collective Vol. I, 503.
Limpricht, *Ann.*, 1898, **299**, 306.

4-*p*-Toluylbenzoic Acid (*4′-Methylbenzophenone-4-carboxylic acid*).

Needles from MeOH. M.p. 228°. Sol. EtOH, Me_2CO. Spar. sol. H_2O, $CHCl_3$, C_6H_6.

Me ester: needles from MeOH. M.p. 126°.

Chloride: $C_{15}H_{11}O_2Cl$. MW, 258·5. Cryst. from pet. ether. M.p. 110°.

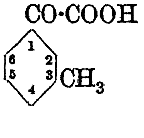
Amide: prisms from EtOH. M.p. 196°.

Limpricht, Claus, *Ann.*, 1900, **312**, 92.

Toluylcatechol.
See Dihydroxymethylbenzophenone.

3-*p*-Toluyl-*p*-cresol.
See 2-Hydroxy-5 : 4′-dimethylbenzophenone.

***m*-Toluylformic Acid** (*m-Tolylglyoxylic acid, α-keto-m-tolylacetic acid, 3-methylbenzoylformic acid*)



$C_9H_8O_3$ MW, 164

Colourless needles from C_6H_6–pet. ether. M.p. 78–82°. B.p. 148–50°/15–16 mm. Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O. Decomp. on dist. under atm. press. ⟶ *m*-toluic acid + *m*-toluic aldehyde. H_2SO_4 + thiophene on C_6H_6 sol. ⟶ red ⟶ bluish-violet col.

Me ester: $C_{10}H_{10}O_3$. MW, 178. B.p. 246–50°/763 mm. slight decomp., 137–8°/11–12 mm.

Et ester: $C_{11}H_{12}O_3$. MW, 192. Oil with pleasant odour. B.p. 250–5° part. decomp., 141–2°/11–12 mm.

Phenylhydrazone: yellow needles from AcOH.Aq. M.p. 158°.

Posner, Heumann, *Ber.*, 1923, **56**, 1624.

***p*-Toluylformic Acid** (*p-Tolylglyoxylic acid, α-keto-p-tolylacetic acid, 4-methylbenzoylformic acid*).

Needles from ligroin. M.p. 97°. B.p. 164°/10 mm. Sol. most org. solvents. Spar. sol. hot H_2O. Dist. at ord. press. ⟶ *p*-toluic acid + *p*-toluic aldehyde. Hot H_2SO_4 ⟶ *p*-toluic acid. Ox. ⟶ *p*-toluic acid + terephthalic acid. Red. ⟶ *p*-tolylglycollic acid + *p*-tolylacetic acid.

Et ester: $C_{11}H_{12}O_3$. MW, 192. Liq. B.p. 260–70°, 154–6°/18 mm. *Phenylhydrazone*: cryst. from ligroin–pet. ether. M.p. 94°.

Amide: $C_9H_9O_2N$. MW, 163. Prisms from EtOH. M.p. 160°.

Nitrile: C_9H_7ON. MW, 145. Prisms. M.p. 92°. Insol. H_2O. *Oxime*: leaflets from H_2O. M.p. 117°.

Phenylhydrazone: yellow needles from C_6H_6–ligroin. M.p. 145–6°.

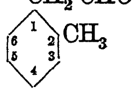
Avogadro, *Gazz. chim. ital.*, 1923, **53**, 698.
Auwers, *Ber.*, 1911, **44**, 600.
Bouveault, *Bull. soc. chim.*, 1897, **17**, 363, 367.

Toluylic Acid.
See Phenylacetic Acid.

Toluylphenol.
See Hydroxy-4-methylbenzophenone.

Toluylresorcinol.
See Dihydroxymethylbenzophenone.

***o*-Tolylacetaldehyde**



$C_9H_{10}O$ MW, 134

Liq. with odour of jasmine. B.p. 219–21°/742 mm., 142–3°/90 mm., 92°/10 mm. D_4^{10} 1·0241. Misc. with EtOH, Et_2O, $CHCl_3$. Spar. misc. with H_2O, C_6H_6, ligroin.

Oxime: needles. M.p. 102–3°. Sol. Et_2O, EtOH, $CHCl_3$. Spar. sol. H_2O, C_6H_6, ligroin.

Spath, *Monatsh.*, 1915, **36**, 8.

***m*-Tolylacetaldehyde.**

Pleasant-smelling, pale yellow oil. B.p. 99–100°/18 mm. Reduces $NH_3.AgNO_3$. Hot alkalis ⟶ resin.

Benzoylhydrazone: needles from EtOH.Aq. M.p. 129–30°.

m-*Nitrobenzoylhydrazone*: yellow needles from EtOH.Aq. M.p. 115–16°.

Curtius, Marangolo, *J. prakt. Chem.*, 1916, **94**, 337.

***p*-Tolylacetaldehyde.**

Needles. M.p. about 40°. B.p. 221–2°, 96°/10 mm., 80–2°/3 mm. D_4^{20} 1·0052. n_D^{20} 1·5255. Very sol. Et_2O. Sol. EtOH, $CHCl_3$. Spar. sol. C_6H_6. Insol. H_2O. Slowly oxidises in air to *p*-tolylacetic acid. Readily decomp. by acids. Electrolytic red. ⟶ β-*p*-tolylethyl alcohol. Forms bisulphite comp. Used in perfumery.

Oxime: prisms from Et_2O. M.p. 126–126·5°. Very sol. Et_2O, $CHCl_3$. Sol. C_6H_6, ligroin. Insol. H_2O.

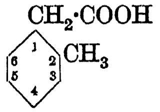
Semicarbazone: cryst. from EtOH. M.p. about 208°.

Knorr, Weissenborn, Laage, U.S.P., 1,899,340, (*Chem. Zentr.*, 1932, II, 2747); D.R.P., 591,452, (*Chem. Zentr.*, 1932, II, 2748).
Spath, *Ber.*, 1914, **47**, 767.
Auwers, *Ber.*, 1906, **39**, 3761.

Tolyl acetate.

See under Cresol.

***o*-Tolylacetic Acid** (2-*Methylphenylacetic acid*)



$C_9H_{10}O_2$ MW, 150

Needles from H_2O. M.p. 88–9°. Very sol. hot H_2O. Ox. ⟶ phthalic acid. Electrolytic red. ⟶ β-*o*-tolylethyl alcohol.

Benzyl ester: b.p. 158–62°/1·5 mm. D_4^{25} 1·0773. n_D^{25} 1·4575.

Amide: $C_9H_{11}ON$. MW, 149. Plates from H_2O. M.p. 161°. Very sol. hot EtOH. Spar. sol. cold H_2O, Et_2O. Sublimes.

Nitrile: *o*-tolubenzyl cyanide, *o*-xylyl cyanide. C_9H_9N. MW, 131. Liq. B.p. 244°. D^{22} 1·0156.

Schorigin, *Ber.*, 1910, **43**, 1941.
Radziszewski, Wispek, *Ber.*, 1885, **18**, 1281.

***m*-Tolylacetic Acid** (3-*Methylphenylacetic acid*).

Needles. M.p. 61°. Very sol. hot H_2O. Electrolytic red. ⟶ β-*m*-tolylethyl alcohol.

Me ester: $C_{10}H_{12}O_2$. MW, 164. B.p. 228–9°. $D^{17·5}$ 1·044.

Et ester: $C_{11}H_{14}O_2$. MW, 178. B.p. 237–8°. $D^{17·5}$ 1·018.

Amide: needles from H_2O. M.p. 141°. Sol. hot EtOH. Spar. sol. cold H_2O, Et_2O. Sublimes in plates.

Nitrile: *m*-tolubenzyl cyanide, *m*-xylyl cyanide. B.p. 240–1°. D^{22} 1·0022.

Radziszewski, Wispek, *Ber.*, 1885, **18**, 1282.
Seńkowski, *Monatsh.*, 1888, **9**, 855.

***p*-Tolylacetic Acid** (4-*Methylphenylacetic acid*).

Needles or plates from H_2O. M.p. 94°. B.p. 265–7°. Sol. hot H_2O, EtOH, Et_2O, $CHCl_3$, C_6H_6. Sublimes. Electrolytic red. ⟶ β-*p*-tolylethyl alcohol.

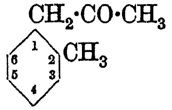
Et ester: liq. with unpleasant odour. B.p. 240°.

Amide: plates from H_2O. M.p. 185°. Sol. hot EtOH. Spar. sol. cold H_2O, Et_2O. Sublimes.

Nitrile: *p*-tolubenzyl cyanide, *p*-xylyl cyanide. F.p. 18°. M.p. 18°. B.p. 242–3°. D^{22} 0·9922.

Schorigin, *Ber.*, 1910, **43**, 1941.
Radziszewski, Wispek, *Ber.*, 1885, **18**, 1281.
Wilds, Meader, *J. Org. Chem.*, 1948, **13**, 763.

***o*-Tolylacetone** (β-*Keto*-α-o-*tolylpropane*, 2-*acetonyltoluene*, *methyl* o-*tolubenzyl ketone*, *methyl* o-*xylyl ketone*)



$C_{10}H_{12}O$ MW, 148

Liq. B.p. 227°, 122°/23 mm.

Oxime: m.p. 75°.

Semicarbazone: m.p. 181°.

Ruzicka, Ehmann, Weisz, *Helv. Chim. Acta*, 1932, **15**, 159.
Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 195.

***m*-Tolylacetone** (β-*Keto*-α-m-*tolylpropane*, 3-*acetonyltoluene*, *methyl* m-*tolubenzyl ketone*, *methyl* m-*xylyl ketone*).

Liq. B.p. 228–9°. D^0 1·019.

Semicarbazone: m.p. 139°.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 195.

***p*-Tolylacetone** (β-*Keto*-α-p-*tolylpropane*, 4-*acetonyltoluene*, *methyl* p-*tolubenzyl ketone*, *methyl* p-*xylyl ketone*).

Liq. B.p. 232–3°, 109–10°/12 mm. Forms bisulphite comp.

Oxime: prisms from pet. ether. M.p. 90–1°.

Semicarbazone: m.p. 158°.

Ruzicka, Ehmann, Rierink, *Helv. Chim. Acta*, 1932, **15**, 160.
Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 195.

ω-*p*-Tolylacetophenone.

See 4-Methyldeoxybenzoin.

p-Tolylacetylene

C_9H_8 MW, 116

Prisms. M.p. 23°. B.p. 168°, 65–7°/18 mm. D^{25} 0·9159. n_D^{25} 1·5447. Gives explosive Cu and Ag derivs.

Otto, *J. Am. Chem. Soc.*, 1934, **56**, 1393.
Vaughn, *ibid.*, 2064.
Gattermann, *Ann.*, 1906, **347**, 359.

2-p-Tolylacrolein.
See p-Methylcinnamaldehyde.

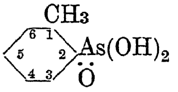
2-Tolylacrylic Acid.
See o-, m-, and p-Methylcinnamic Acid.

Tolylaminophenol.
See Hydroxy-methyldiphenylamine.

Tolylanisidine.
See under Hydroxy-methyldiphenylamine.

N-Tolylanthranilic Acid.
See Methyldiphenylamine-carboxylic Acid.

o-Tolylarsinic Acid (o-*Toluenearsonic acid*)



$C_7H_9O_3As$ MW, 216

Needles from H_2O. M.p. 159–60°. Sol. H_2O, EtOH. Loses H_2O at m.p.

Ruddy, Starkey, *Organic Syntheses*, 1946, XXVI, 62.

m-Tolylarsinic Acid (m-*Toluenearsonic acid*).

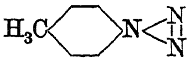
Needles from H_2O. M.p. 150°.

Ruddy, Starkey, *Organic Syntheses*, 1946, XXVI, 62.

p-Tolylarsinic Acid (p-*Toluenearsonic acid*).
Needles from H_2O. Loses H_2O at 105–10°.

Ruddy, Starkey, *Organic Syntheses*, 1946, XXVI, 62.

p-Tolyl azide (p-*Azidotoluene*)



$C_7H_7N_3$ MW, 133

Yellow oil. B.p. 80°/10 mm. Decomp. at 180°. D_4^{23} 1·0527. Volatile in steam.

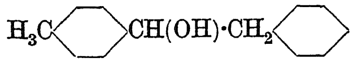
Lindemann, Thiele, *Ber.*, 1928, **61**, 1529.
Ponzio, *Gazz. chim. ital.*, 1916, **46**, 57.
Dimroth, Pfister, *Ber.*, 1910, **43**, 2760.

Tolylazoimide.
See Methylbenztriazole.

Tolylbenzoic Acid.
See Methyldiphenyl-carboxylic Acid.

Tolylbenzylamine.
See N-Benzyltoluidine.

p-Tolylbenzylcarbinol (β-*Phenyl*-α-p-*tolylethyl alcohol*, α-*hydroxy*-4-*methyldiphenylethane*)



$C_{15}H_{16}O$ MW, 212

Needles. M.p. 107–8° (66°). Dist. without decomp. above 360°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6.

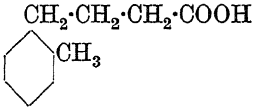
Tiffeneau, Lévy, *Bull. soc. chim.*, 1931, **49**, 1738.
Mann, *Ber.*, 1881, **14**, 1646.

Tolyl benzyl Ketone.
See 2′-, 3′, *and* 4′-Methyldeoxybenzoin.

Tolylbutane.
See Butyltoluene.

Tolylbutenone.
See Methyl methylstyryl Ketone.

3-o-Tolylbutyric Acid



$C_{11}H_{14}O_2$ MW, 178

M.p. 70·5°.
Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 140 50°/9 mm.

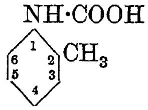
Wessely, Wang, *Ber.*, 1940, **73**, 19.

2-p-Tolylbutyric Acid.
See Curcumic Acid.

3-p-Tolylbutyric Acid.
M.p. 61–2°. B.p. 124–34°/0·3–0·5 mm.

Martin, *J. Am. Chem. Soc.*, 1936, **58**, 1438.

o-Tolylcarbamic Acid (o-*Methylcarbanilic acid*)



$C_8H_9O_2N$ MW, 151

β-*Chloroethyl ester*: $C_{10}H_{12}O_2NCl$. MW, 213·5. Needles from C_6H_6. M.p. 45°. B.p. 209–10°/37 mm.
γ-*Chloropropyl ester*: $C_{11}H_{14}O_2NCl$. MW, 227·5. Needles from ligroin. M.p. 49° (46–46·5°). B.p. 182·5°/4·5 mm. (170–5°/5 mm.).
Butyl ester: $C_{12}H_{17}O_2N$. MW, 207. Prisms. M.p. 45·5°.

Chattaway, Saerens, *J. Chem. Soc.*, 1920, **117**, 711.
Adams, Segur, *J. Am. Chem. Soc.*, 1923, **45**, 787.
Adams, Pierce, *ibid.*, 793.

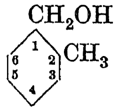
p-Tolylcarbamic Acid (p-*Methylcarbanilic acid*).
Et ester: *p*-tolylurethane. $C_{10}H_{13}O_2N$. MW, 179. B.p. 243–7°, 128–32°/12 mm.
Butyl ester: prisms. M.p. 63°.

β-*Chloroethyl ester*: cryst. from C_6H_6. M.p. 61°.

γ-*Chloropropyl ester*: straw-coloured oil. B.p. 188°/4·5 mm. D_{20}^{20} 1·186. n_D^{18} 1·494.

Chattaway, Saerens, *J. Chem. Soc.*, 1920, **117**, 711.
Adams, Segur, *J. Am. Chem. Soc.*, 1923, **45**, 787.
Adams, Pierce, *ibid.*, 793.

***o*-Tolylcarbinol** (2-*Methylbenzyl alcohol*)



$C_8H_{10}O$ MW, 122

Needles. M.p. 36°. B.p. 219°, 135°/30 mm. D^{40} 1·023. Sol. 100 parts cold H_2O, 60 parts at the boil. Very sol. EtOH, Et_2O, $CHCl_3$. Ox. ⟶ *o*-toluic aldehyde.

Me ether: $C_9H_{12}O$. MW, 136. Oil with unpleasant odour. B.p. 187–8°.

Et ether: $C_{10}H_{14}O$. MW, 150. Sweet-smelling oil. B.p. 208–10°.

Isoamyl ether: $C_{13}H_{20}O$. MW, 192. Oil with aromatic odour. B.p. 125°/15 mm.

Acetyl: liq. B.p. 228–30°/753 mm.

p-*Nitrobenzoyl*: cryst. from MeOH. M.p. 100–1°.

Urethane: m.p. 79°.

Colson, *Ann. chim.*, 1885, **6**, 115.
Smith, Spillane, *J. Am. Chem. Soc.*, 1940, **62**, 2639.

***m*-Tolylcarbinol** (3-*Methylbenzyl alcohol*). Oil. B.p. 217°. D^{17} 0·9157. Sol. 20 parts cold H_2O. Misc. with EtOH, Et_2O. Ox. ⟶ *m*-toluic aldehyde + *m*-toluic acid.

Et ether: b.p. 202°/740 mm. D^{17} 0·9302.

Acetyl: oil. B.p. 226°.

Colson, *Ann. chim.*, 1885, **6**, 117.
Radziszewski, Wispek, *Ber.*, 1882, **15**, 1747.

***p*-Tolylcarbinol** (4-*Methylbenzyl alcohol*). Needles from heptane. M.p. 60°. B.p. 217°, 116–18°/20 mm. Sol. EtOH, Et_2O. Spar. sol. cold H_2O, mod. sol. hot.

Et ether: b.p. 203°/740 mm. D^{17} 0·9304.

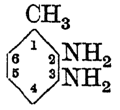
Acetyl: oil. B.p. 220°.

3 : 5-*Dinitrobenzoyl*: m.p. 117–18°.

Oddo, *Gazz. chim. ital.*, 1911, **41**, 285.
Cannizzaro, *Ann.*, 1862, **124**, 255.
Mozingo, Folkers, *J. Am. Chem. Soc.*, 1948, **70**, 229.
Davidson, Weiss, *Organic Syntheses*, 1938, XVIII, 79.

2-*p*-Tolylcinchoninic Acid.
See 2-*p*-Tolylquinoline-4-carboxylic Acid.

2:3-Tolylenediamine (2:3-*Diaminotoluene*, 3-*methyl*-o-*phenylenediamine*)



$C_7H_{10}N_2$ MW, 122

Cryst. M.p. 63–4°. B.p. 255°. Sol. most. org. solvents. Boiling H·COOH ⟶ 4-methylbenziminazole.

Gabriel, Thieme, *Ber.*, 1919, **52**, 1081.

2:4-Tolylenediamine (2:4-*Diaminotoluene*, 4-*methyl*-m-*phenylenediamine*, unsym.-m-*toluylenediamine*).

Needles from H_2O, prisms from EtOH. M.p. 99°. B.p. 292°, 148–50°/8 mm. Very sol. boiling H_2O, EtOH, Et_2O. Aq. sol. darkens in air. Reduces warm $NH_3.AgNO_3$. Ox. ⟶ 2-amino-4-nitrotoluene + 2 : 4-dinitrotoluene. Sol. in dil. HCl on warming with dil. $KClO_3$ ⟶ light violet col.

2-N-*Me*: $C_8H_{12}N_2$. MW, 136. Oil. B.p. 273°. 2-N-*Benzoyl*: needles. M.p. 167°.

2-N-*Di-Me*: *see* 2-*N*-Dimethyl-2 : 4-tolylenediamine.

4-N-*Di-Me*: *see* 4-*N*-Dimethyl-2 : 4-tolylenediamine.

N-*Tetra-Me*: $C_{11}H_{18}N_2$. MW, 178. Brownish-yellow oil. B.p. 255–6°/757 mm., 148–50°/24–6 mm. D^{24} 0·9661. *Picrate*: prisms from AcOEt. M.p. 162–3°.

2-N-*Et*: $C_9H_{14}N_2$. MW, 150. Liq. B.p. 274–5°.

4-N-*Et*: liq. B.p. 289–91°.

2-N-*Di-Et*: $C_{11}H_{18}N_2$. MW, 178. Yellow oil. B.p. 259°. Volatile in steam. *B,2HCl*: cryst. + H_2O. M.p. 213–15°.

4-N-*Butyl*: $C_{11}H_{18}N_2$. MW, 178. Needles from Et_2O. M.p. 53°. Spar. sol. H_2O, ligroin.

2-N-*Phenyl*: *see* 5-Amino-2-methyldiphenylamine.

4-N-*Phenyl*: *see* 3-Amino-4-methyldiphenylamine.

4-N-*Et*-4-*Phenyl*: $C_{15}H_{18}N_2$. MW, 226. Cryst. from ligroin. M.p. 59–60°.

4-N-p-*Tolyl*: *see* 3-Amino-4 : 4′-dimethyldiphenylamine.

2-N-*Benzyl*: $C_{14}H_{16}N_2$. MW, 212. Needles. M.p. 81°. Very sol. EtOH, Et_2O. Insol. cold H_2O.

4-N-*Formyl*: cryst. from H_2O. M.p. 113–14°.

2 : 4-N-*Diformyl*: needles from H_2O. M.p. 176–7°.

2-N-*Acetyl*: needles. M.p. 140°.

4-N-*Acetyl*: prisms or needles. M.p. 161·5°.

2 : 4-N-*Diacetyl*: needles. M.p. 224°.

4-N-*Benzoyl*: prisms. M.p. 142°.

2 : 4-N-*Dibenzoyl*: plates from AcOH. M.p. 224°.

2-N-*Benzenesulphonyl*: plates from EtOH. M.p. 138°.

4-N-*Benzenesulphonyl*: m.p. 138°.
2 : 4-N-*Dibenzenesulphonyl*: needles. M.p. 191°.
4-N-p-*Toluenesulphonyl*: cryst. powder. M.p. 160°.
2 : 4-N-*Di*-p-*toluenesulphonyl*: needles. M.p. 192–3°.

Mahood, Schaffner, *Organic Syntheses*, 1931, XI, 32.
Gnehm, Blumer, *Ann.*, 1899, **304**, 106.

2 : 5-Tolylenediamine (2:5-*Diaminotoluene*, 2-*methyl*-p-*phenylenediamine*, p-*toluylenediamine*).
Plates from C_6H_6. M.p. 64°. B.p. 273–4°. Sol. H_2O, EtOH, Et_2O, hot C_6H_6. Spar. sol. cold C_6H_6. $MnO_2 + H_2SO_4 \longrightarrow$ toluquinone.
2-N-*Me*: $C_8H_{12}N_2$. MW, 136. Thick oil. B.p. 276–276·5°. Rapidly oxidises in air. 2-p-*Toluenesulphonyl*: plates from EtOH.Aq. M.p. 118–19°.
2-N-*Di-Me*: *see* 2-*N*-Dimethyl-2 : 5-tolylenediamine.
5-N-*Di-Me*: *see* 5-*N*-Dimethyl-2 : 5-tolylenediamine.
2 : 5-N-*Tetra-Me*: $C_{11}H_{18}N_2$. MW, 178. Oil. B.p. 260°. $FeCl_3 \longrightarrow$ blue col. *Monomethiodide*: needles from H_2O. M.p. 160°.
2-N-*Et*: $C_9H_{14}N_2$. MW, 150. Thick oil. B.p. 272° (in hydrogen). *B,2HCl*: m.p. 124° decomp. 5-N-*Benzoyl*: needles from C_6H_6–ligroin. M.p. 174°.
2-N-*Di-Et*: $C_{11}H_{18}N_2$. MW, 178. M.p. 24°. B.p. 266–7°.
2-N-*Phenyl*: *see* 4-Amino-2-methyldiphenylamine.
5-N-*Phenyl*: *see* 4-Amino-3-methyldiphenylamine.
5-N-o-*Tolyl*: *see* 4-Amino-3 : 2′-dimethyldiphenylamine.
2 : 5-N-*Di*-p-*tolyl*: $C_{21}H_{22}N_2$. MW, 302. Plates from AcOH. M.p. 112–13°.
2 : 5-N-*Diacetyl*: prisms from EtOH.Aq. M.p. 220°.
2-N-*Benzenesulphonyl*: needles from H_2O. M.p. 147°.
2-N-p-*Toluenesulphonyl*: prisms from EtOH.Aq. M.p. 150°.

Nietzki, *Ber.*, 1877, **10**, 1157.

2 : 6-Tolylenediamine (2:6-*Diaminotoluene*, 2-*methyl*-m-*phenylenediamine*).
Prisms from H_2O. M.p. 105°. $FeCl_3 \longrightarrow$ deep brown col.
N-*Diacetyl*: m.p. 202–3°.

Ullmann, *Ber.*, 1884, **17**, 1959.

3 : 4-Tolylenediamine (3:4-*Diaminotoluene*, 4-*methyl*-o-*phenylenediamine*, unsym.-o-*toluylenediamine*).
Plates from ligroin. M.p. 89–90°. B.p. 265°. Mod. sol. cold H_2O. Sublimes. Base and salts rapidly oxidise in aq. sol. in air. Boiling H·COOH $\longrightarrow$ 5-methylbenziminazole.
4-N-*Me*: $C_8H_{12}N_2$. MW, 136. Plates from Et_2O. M.p. 43–4°. B.p. 260°/752 mm. *B,HCl*: plates from EtOH. M.p. 175–80°. *B,2HCl*: prisms from EtOH. M.p. 175–85° decomp. $(B,COOH)_2$: needles. M.p. 124°. *Picrate*: yellow cryst. M.p. 164°. 3 : 4-N-*Diacetyl*: plates from H_2O. M.p. 183–4°. 4-N-p-*Toluenesulphonyl*: needles. M.p. 133°.
3 : 4-N-*Di-Me*: $C_9H_{14}N_2$. MW, 150. Oil. B.p. 259–60°/740 mm. $FeCl_3 \longrightarrow$ red col. *B,2HCl*: needles. M.p. 125°.
4-N-*Di-Me*: *see* 4-*N*-Dimethyl-3 : 4-tolylenediamine.
3 : 4-N-*Tetra-Me*: $C_{11}H_{18}N_2$. MW, 178. Liq. B.p. 224·5–225·5°/717 mm.
3-N-*Et*: $C_9H_{14}N_2$. MW, 150. Needles from pet. ether. M.p. 59°. Becomes violet in air. $FeCl_3 \longrightarrow$ red col.
4-N-*Et*: plates from CS_2.Aq. M.p. 55°. Very sol. most org. solvents. Not very stable. *B,HCl*: cryst. from EtOH. M.p. 176°. $B_2,(COOH)_2$: needles. M.p. 151°. 3-N-*Acetyl*: cryst. from EtOH. M.p. 177°.
3 : 4-N-*Di-Et*: *see sym.*-Diethyl-3 : 4-tolylenediamine.
4-N-*Butyl*: $C_{11}H_{18}N_2$. MW, 178. Very readily oxidises. 4-N-*Acetyl*: needles from Et_2O–pet. ether. M.p. 102°. 3 : 4-N-*Diacetyl*: cryst. from EtOH.Aq. M.p. 130°.
3-N-*Phenyl*: *see* 6-Amino-3-methyldiphenylamine.
4-N-*Phenyl*: *see* 2-Amino-4-methyldiphenylamine.
3-N-p-*Tolyl*: *see* 6-Amino-3 : 4′-dimethyldiphenylamine.
4-N-p-*Tolyl*: *see* 2-Amino-4 : 4′-dimethyldiphenylamine.
3-N-*Acetyl*: cryst. from $CHCl_3$–pet. ether. M.p. about 95°.
4-N-*Acetyl*: needles. M.p. 131–2°.
3 : 4-N-*Diacetyl*: needles from H_2O. M.p. 210°.
3-N-*Benzoyl*: needles from C_6H_6. M.p. 158°.
4-N-*Benzoyl*: needles from EtOH or C_6H_6. M.p. 193–4°.
3 : 4-N-*Dibenzoyl*: needles from AcOH. M.p. 263–4°.
3-N-*Benzenesulphonyl*: needles from C_6H_6. M.p. 134–5°.
4-N-*Benzenesulphonyl*: needles from EtOH.Aq. M.p. 146–7°.
3 : 4-N-*Dibenzenesulphonyl*: plates from EtOH. M.p. 178–9°.
4-N-p-*Toluenesulphonyl*: needles. M.p. 140°.

Reilly, Hickinbottom, *J. Chem. Soc.*, 1919, **115**, 177.
Fischer, *Ber.*, 1893, **26**, 194.
Bamberger, Wulz, *Ber.*, 1891, **24**, 2082.
Noelting, Stoecklin, *ibid.*, 565.

3 : 5-Tolylenediamine (*3 : 5-Diaminotoluene, 5-methyl-*m*-phenylenediamine,* sym.-m-*toluylenediamine*).

Oil. B.p. 283–5°. Very sol. H_2O.

B,2HCl : needles. M.p. 255–60° decomp.

3 : 5-N-*Diphenyl* : $C_{19}H_{18}N_2$. MW, 274. Cryst. from AcOH. M.p. 105°. Sol. cold EtOH, Et_2O, CS_2, C_6H_6. 3 : 5-N-*Diacetyl* : needles. M.p. 160°. 3 : 5-N-*Dibenzoyl* : needles from C_6H_6–ligroin. M.p. 190–1°.

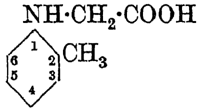
3 : 5-N-*Diacetyl* : prisms from EtOH. M.p. 235–6°.

Davis, *J. Chem. Soc.*, 1902, **81**, 873.

1-Tolylethyl Alcohol.
See Methyltolylcarbinol.

Tolylethylene.
See Methylstyrene.

***o*-Tolylglycine** (o-*Toluidinoacetic acid*)



$C_9H_{11}O_2N$ MW, 165

Needles from EtOH. M.p. 149–50° (160°). Very sol. EtOH, Et_2O. Insol. cold H_2O. $k = 5{\cdot}9 \times 10^{-5}$ at 25°. Reduces silver salts.

Et ester : $C_{11}H_{15}O_2N$. MW, 193. Cryst. M.p. 26°. B.p. 280°. D^{20} 1·058.

Amide : $C_9H_{12}ON_2$. MW, 164. Needles from H_2O. M.p. 140°. Sol. EtOH, hot H_2O.

N-*Et* : $C_{11}H_{15}O_2N$. MW, 193. Cryst. from C_6H_6. M.p. 63–4°.

N-*Formyl* : cryst. from H_2O. M.p. 113–15°.

N-*Acetyl* : plates from EtOH.Aq. M.p. 210–12°. $k = 2{\cdot}19 \times 10^{-4}$ at 25°.

N-*Chloroacetyl* : plates from C_6H_6. M.p. 116–17°.

N-*Bromoacetyl* : plates from H_2O. M.p. 124°.

Staats, *Ber.*, 1880, **13**, 137.
Ehrlich, *Ber.*, 1883, **16**, 204.
Steppes, *J. prakt. Chem.*, 1900, **62**, 491.

***m*-Tolylglycine** (m-*Toluidinoacetic acid*).

Et ester : plates from EtOH. M.p. 68°. Sol. EtOH, Et_2O, AcOH. Very spar. sol. hot H_2O.

Ehrlich, *Ber.*, 1882, **15**, 2011.
Gault, *Bull. soc. chim.*, 1908, **3**, 372.

***p*-Tolylglycine** (p-*Toluidinoacetic acid*).

Plates from Et_2O–pet. ether. M.p. 132° (120–1°). Insol. pet. ether, cold H_2O. Unstable in air. $k = 1{\cdot}5 \times 10^{-5}$ at 25°. Reduces Tollen's reagent.

Et ester : m.p. 52–3° (48–9°). B.p. 279° slight decomp. Very sol. Et_2O. Mod. sol. cold EtOH. Very spar. sol. hot H_2O.

Amide : cryst. from H_2O. M.p. 168° (162–3°). Sol. EtOH, C_6H_6, hot H_2O. Insol. cold H_2O.

Nitrile : $C_9H_{10}N_2$. MW, 146. Cryst. M.p. 62°. Very sol. common org. solvents except ligroin. N-*Me* : m.p. 57°. B.p. 156–7°/9 mm.

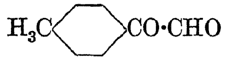
Anilide : needles from H_2O. M.p. 82–3°.

N-*Acetyl* : plates from H_2O. M.p. 175–6°.

Steppes, *J. prakt. Chem.*, 1900, **62**, 487.
Bischoff, Hausdörfer, *Ber.*, 1892, **25**, 2282.
v. Miller, Plöchl, Sieber, *Ber.*, 1898, **31**, 2715.
M.L.B., D.R.P., 175,797, (*Chem. Zentr.*, 1906, II, 1700).

Tolylglycollic Acid.
See Methylmandelic Acid.

***p*-Tolylglyoxal** (p-*Toluylformaldehyde*)



$C_9H_8O_2$ MW, 148

Needles + $1H_2O$. M.p. anhyd. 111–12° (101°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O, ligroin.

Aldoxime : needles from C_6H_6. M.p. 100°. *Acetyl* : plates from MeOH. M.p. 67–8°.

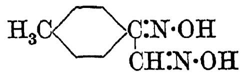
Phenylhydrazone : plates from EtOH. M.p. 165°.

Dioxime : *see* *p*-Tolylglyoxime.

Phenylosazone : yellow needles from EtOH. M.p. 145°.

Müller, v. Pechmann, *Ber.*, 1889, **22**, 2560.
Neuberg, Ostendorf, *Biochem. Z.*, 1935, **279**, 459.

***p*-Tolylglyoxime**



$C_9H_{10}O_2N_2$ MW, 178

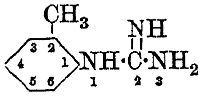
α-Form :
M.p. 170–1°. Above m.p. ⟶ β-form.
Diacetyl : m.p. 115°.
Monobenzyl : m.p. 147–8°.

β-Form :
M.p. 192–3°. Forms Ni salt.
Diacetyl : m.p. 73–4°.
Dibenzoyl : m.p. 170°.

Avogadro, *Gazz. chim. ital.*, 1923, **53**, 698.

Tolylglyoxylic Acid.
See Toluylformic Acid.

***o*-Tolylguanidine** (*Guanyl-*o*-toluidine*)



$C_8H_{11}N_3$ MW, 149

B,HCl : cryst. from Me_2CO–Et_2O. M.p. 133–5°. Sol. H_2O, Me_2CO. Insol. Et_2O.

Nitrate : cryst. from H_2O. M.p. 133°. Very bitter taste.

Picrate : m.p. 223–4°.

3-*Phenyl* : *see* Phenyl-*o*-tolylguanidine.

Braun, *J. Am. Chem. Soc.*, 1933, **55**, 1281.
M.L.B., D.R.P., 172,979, (*Chem. Zentr.*, 1906, II, 984).

m-Tolylguanidine (*Guanyl*-m-*toluidine*).

Sulphate: cryst. from EtOH–Et_2O. M.p. 215–17°. Sol. H_2O. Insol. Et_2O.

Braun, *J. Am. Chem. Soc.*, 1933, **55**, 1282.

p-Tolylguanidine (*Guanyl*-p-*toluidine*).

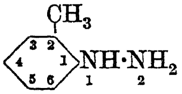
B,HCl: cryst. from EtOH–Et_2O. M.p. 136–7°. Sol. H_2O, EtOH. Insol. Et_2O.

B,HNO$_3$: m.p. 146–7°. Very bitter taste.

3-*Phenyl*: *see* Phenyl-*p*-tolylguanidine.

Braun, *J. Am. Chem. Soc.*, 1933, **55**, 1282.
Kämpf, *Ber.*, 1904, **37**, 1683.

o-Tolylhydrazine (o-*Hydrazinotoluene*)



$C_7H_{10}N_2$ MW, 122

Needles. M.p. 59° (56°). Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. cold ligroin.

B,HNO$_3$: m.p. 98–100° (sinters about 75°).

2-N-*Phenyl*: *see* 2-Methylhydrazobenzene.

2-N-*Formyl*: plates from H_2O. M.p. 120°.

2-N-*Acetyl*: leaflets from H_2O. M.p. 104°.

2-N-*Propionyl*: plates from H_2O. M.p. 83–4°.

2-N-*Isobutyryl*: leaflets from C_6H_6–pet. ether. M.p. 93°.

2-N-*Benzoyl*: needles. M.p. 180°.

Fischer, Bösler, *Ann.*, 1882, **212**, 338.
Gallinek, v. Richter, *Ber.*, 1885, **18**, 3175.

m-Tolylhydrazine (m-*Hydrazinotoluene*).

Oil. B.p. 240–4°.

B,HNO$_3$: needles. M.p. 145–7°.

2-N-*Phenyl*: *see* 3-Methylhydrazobenzene.

2-N-*Propionyl*: m.p. 131°.

Buchka, Schachtebeck, *Ber.*, 1889, **22**, 841.

p-Tolylhydrazine (p-*Hydrazinotoluene*).

Leaflets from Et_2O. M.p. 65–6° (61°). B.p. 240–4° slight decomp. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O.

B,HNO$_3$: leaflets. M.p. 152–3°.

2-N-*Phenyl*: *see* 4-Methylhydrazobenzene.

2-N-*Formyl*: prisms or needles from EtOH. M.p. 166·5°.

2-N-*Acetyl*: cryst. M.p. 130° (127°).

2-N-*Propionyl*: needles from H_2O. M.p. 170°.

2-N-*Isobutyryl*: leaflets. M.p. 147–8°.

1-N-*Benzoyl*: leaflets from C_6H_6. M.p. 68–70°. 2-N-*Acetyl*: needles from C_6H_6. M.p. 135°.

2-N-*Benzoyl*: leaflets from C_6H_6. M.p. 143°.

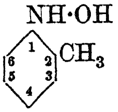
1 : 2-N-*Dibenzoyl*: leaflets from EtOH. M.p. 188°.

McPherson, Stratton, *J. Am. Chem. Soc.*, 1915, **37**, 908.
Bamberger, *Ber.*, 1898, **31**, 582.
Fischer, *Ber.*, 1876, **9**, 890.

Tolyl α-hydroxybenzyl Ketone.

See Methylbenzoin.

N-o-Tolylhydroxylamine (o-*Hydroxylaminotoluene*)



C_7H_9ON MW, 123

Needles from Et_2O–C_6H_6. M.p. 44°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. ligroin.

Bamberger, Rising, *Ann.*, 1901, **316**, 278.
Bretschneider, *J. prakt. Chem.*, 1897, **55**, 293.

N-m-Tolylhydroxylamine (m-*Hydroxylaminotoluene*).

Leaflets from C_6H_6–pet. ether. M.p. 68·5°. Sol. EtOH, Et_2O, $CHCl_3$, hot C_6H_6. Mod. sol. hot H_2O. Spar. sol. ligroin.

Bamberger, Rising, *Ann.*, 1901, **316**, 283.

N-p-Tolylhydroxylamine (p-*Hydroxylaminotoluene*).

Leaflets from C_6H_6. M.p. 94°. Sol. EtOH, Et_2O, $CHCl_3$, hot C_6H_6. Insol. cold ligroin. At 115–20° ⟶ *p*-azoxytoluene.

Willstätter, Kubli, *Ber.*, 1908, **41**, 1937.
Bamberger, Rising, *Ann.*, 1901, **316**, 280.

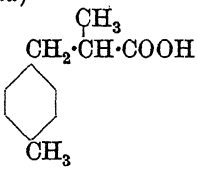
p-Tolyl 5-hydroxy-m-tolyl Ketone.

See 2-Hydroxy-5 : 4′-dimethylbenzophenone.

Tolylisobutylamine.

See *N*-Isobutyl-toluidine.

2-p-Tolylisobutyric Acid (α : 4-*Dimethylhydrocinnamic acid*)



$C_{11}H_{14}O_2$ MW, 178

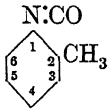
Plates from H_2O. M.p. 85° (174–5° from 90% AcOH). B.p. 168–9°/9 mm.

Et ester: $C_{13}H_{18}O_2$. MW, 206. B.p. 130–2°/10 mm.

Amide: $C_{11}H_{15}ON$. MW, 177. Needles from H_2O. M.p. 130°.

Ruzicka, Ehmann, Delbes, *Helv. Chim. Acta*, 1932, **15**, 162.
Dev, *Chem. Abstracts*, 1948, **42**, 5890.

o-Tolyl isocyanate (o-*Tolylcarbonimide*)



C_8H_7ON MW, 133

Liq. B.p. 185–6°.

Gattermann, Cantzler, *Ber.*, 1892, **25**, 1086.
Haager, Doht, *Monatsh.*, 1906, **27**, 271.

m-Tolyl isocyanate (m-*Tolylcarbonimide*).
B.p. 195–8° (183°). *m*-Toluidine ⟶ di-*m*-tolylurea.

Haager, Doht, *Monatsh.*, 1906, **27**, 273.
Gattermann, Cantzler, *Ber.*, 1892, **25**, 1089.

p-Tolyl isocyanate (p-*Tolylcarbonimide*).
Liq. B.p. 187°/751 mm.

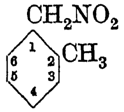
Haager, Doht, *Monatsh.*, 1906, **27**, 273.
Kühn, Henschel, *Ber.*, 1888, **21**, 505 (*Note*).

m-Tolylisopropyl Alcohol.
See Methyl-*m*-xylylcarbinol.

Tolylnaphthylamine.
See under Naphthylamine.

Tolyl naphthyl sulphide.
See under Thionaphthol.

o-Tolylnitromethane (ω-*Nitro*-o-*xylene*)



$C_8H_9O_2N$ MW, 151

M.p. 12–14°. B.p. 145–6°/23 mm., slight decomp., 138–9°/20 mm. D_0^{18} 1·1423. n_D^{18} 1·5439. Turns red on standing.

Konowalow, *Chem. Zentr.*, 1905, II, 817.
Wislicenus, Wren, *Ber.*, 1905, **38**, 503.

m-Tolylnitromethane (ω-*Nitro*-m-*xylene*).
Yellowish liq. B.p. 140°/35 mm. decomp., 128–32°/19 mm. D_0^0 1·1370.

Konowaloff, *Chem. Zentr.*, 1899, I, 1238.
Heilmann, *Ber.*, 1890, **23**, 3165.
Wislicenus, Wren, *Ber.*, 1905, **38**, 505.

p-Tolylnitromethane (ω-*Nitro*-p-*xylene*).
M.p. 11–12°. B.p. 150–1°/35 mm. slight decomp. D_0^{20} 1·1234. n_D^{20} 1·53106.

Konowaloff, *Chem. Zentr.*, 1899, I, 1238.
Wislicenus, Wren, *Ber.*, 1905, **38**, 506.

Tolylphenetidine.
See under Hydroxy-methyldiphenylamine.

4-p-Tolylphenol.
See 4′-Hydroxy-4-methyldiphenyl.

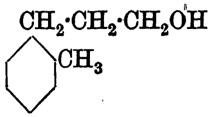
Tolylphenylenediamine.
See Amino-methyldiphenylamine.

2-Tolylpropionaldehyde.
See o- and p-Methylhydrocinnamaldehyde.

1-p-Tolylpropionic Acid.
See p-Methylhydratropic Acid.

2-Tolylpropionic Acid.
See o-, m- and p-Methylhydrocinnamic Acid.

3-o-Tolylpropyl Alcohol (γ-*Hydroxy*-o-*propyltoluene*)



$C_{10}H_{14}O$ MW, 150

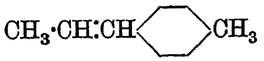
B.p. 136°/15 mm.
Phenylurethane: needles from pet. ether. M.p. 58°.

Harvey, Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 428.

3-m-Tolylpropyl Alcohol (γ-*Hydroxy*-m-*propyltoluene*).
B.p. 140°/14 mm. D_4^{20} 0·9609.
Acetyl: b.p. 136°/10 mm.

v. Braun, Grabowski, Kirschbaum, *Ber.*, 1913, **46**, 1274.

1-p-Tolylpropylene (1-*Methyl*-4-*propenylbenzene*, β : 4-*dimethylstyrene*, p-*propenyltoluene*)

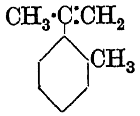


$C_{10}H_{12}$ MW, 132

B.p. 195–7°, 92–3°/20 mm., 83–5°/10 mm. D^{13} 0·9057.
Nitrosochloride: needles. M.p. 135°.

Klages, *Ber.*, 1902, **35**, 2254.

2-o-Tolylpropylene (1-*Methyl*-2-*isopropenylbenzene*, o-*isopropenyltoluene*, α : 2-*dimethylstyrene*)



$C_{10}H_{12}$ MW, 132

B.p. 172–3°. D^0 0·9076. Oxidises in air.

Tiffeneau, *Ann. chim.*, 1907, **10**, 194.

2-m-Tolylpropylene (1-*Methyl*-3-*isopropenylbenzene*, m-*isopropenyltoluene*, α : 3-*dimethylstyrene*).
B.p. 185–6°. D^0 0·9115. Oxidises in air.

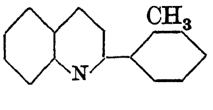
Tiffeneau, *Ann. chim.*, 1907, **10**, 194.

2-p-Tolylpropylene (1-*Methyl*-4-*isopropenylbenzene*, p-*isopropenyltoluene*, α : 4-*dimethylstyrene*).
M.p. – 20°. B.p. 184–5°. D^0 0·9122. Oxidises in air. Slowly polymerises.
Nitrosochloride: cryst. from MeOH. M.p. 100–2°.

Tiffeneau, *Ann. chim.*, 1907, **10**, 194.
Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 653.

1-Tolyl-2-pyridylethylene.
See Methylstyrylpyridine.

2-o-Tolylquinoline



$C_{16}H_{13}N$ MW, 219

Cryst. from Et_2O. M.p. 76·0–76·2°. B.p. 197°/4 mm.
Picrate: m.p. 176°.

Oldham, Johns, *J. Am. Chem. Soc.*, 1939, **61**, 3289.

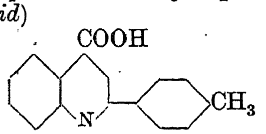
2-*m*-Tolylquinoline (*ψ-Flavoline*).
Needles from C_6H_6–ligroin. M.p. 77°.

Weidel, Bamberger, *Monatsh.*, 1888, **9**, 109.

2-*p*-Tolylquinoline.
Pale yellow plates from EtOH.Aq. M.p. 83°. B.p. 240°/15 mm.
Picrate: m.p. 194°.
Methiodide: decomp. at 192°.
Methosulphate: needles from EtOH–Et_2O. M.p. 158–9°.
Methopicrate: m.p. 157–8°.

Le Fèvre, Le Fèvre, Pearson, *J. Chem. Soc.*, 1934, 41.
v. Braun, Brauns, *Ber.*, 1927, **60**, 1255.

2-*p*-Tolylquinoline-4-carboxylic Acid (*4′-Methylcinchophene, 4′-methylatophan*, 2-p-*tolylcinchonic acid*)

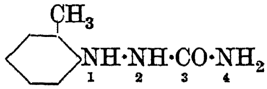


$C_{17}H_{13}O_2N$ MW, 263

Cryst. from EtOH. M.p. 211°. Loses CO_2 at 250°.
Me ester: $C_{18}H_{15}O_2N$. MW, 277. Cryst. M.p. 101°.
Et ester: $C_{19}H_{17}O_2N$. MW, 291. Yellow needles from 70% EtOH. M.p. 54°. Sol. most org. solvents.
Propyl ester: $C_{20}H_{19}O_2N$. MW, 305. Yellow cryst. from EtO_2. M.p. 32°. Sol. usual solvents.
2-Chloroethyl ester: $C_{19}H_{16}O_2NCl$. MW, 325·5. Needles from 70% MeOH. M.p. 79°.
Chloride: $C_{17}H_{12}ONCl$. MW, 281·5. *B,HCl*: cryst. M.p. 188°.
Amide: $C_{17}H_{14}ON_2$. MW, 262. Needles from EtOH. M.p. 208°. Sol. cold Me_2CO. Less sol. C_6H_6, toluene.
Hydrazide: cryst. from chlorobenzene. M.p. 232–3°. Sol. propyl alcohol, amyl alcohol, chlorobenzene. Less sol. MeOH, EtOH, isopropyl alcohol, C_6H_6, toluene, xylene. Insol. Et_2O. *Picrate*: prisms from EtOH. Does not melt below 300°.

v. Braun, Brauns, *Ber.*, 1927, **60**, 1255.
John, Ottawa, *J. prakt. Chem.*, 1931, **131**, 314.
Du Puis, Lindwall, *J. Am. Chem. Soc.*, 1934, **56**, 471.

1-*o*-Tolylsemicarbazide



$C_8H_{11}ON_3$ MW, 165

Needles. M.p. 159–60°. Mod. sol. H_2O. Insol. Et_2O, C_6H_6.
4-N-*Me*: m.p. 158–9°.
4-N-*Et*: needles from EtOH. M.p. 130–1°.
4-N-*Phenyl*: needles from EtOH. M.p. 142°.

Pinner, *Ber.*, 1888, **21**, 1221.

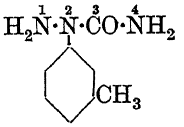
1-*m*-Tolylsemicarbazide.
Leaflets from H_2O or EtOH.Aq. M.p. 183–4°. Insol. Et_2O. Employed as antipyretic under name of Maretin.
4-N-*Phenyl*: needles from EtOH. M.p. 159°.

Bayer, D.R.P., 157,572, (*Chem. Zentr.*, 1905, I, 196).
Arreguine, *Chem. Abstracts*, 1945, **39**, 1963.

1-*p*-Tolylsemicarbazide.
Leaflets or needles. M.p. 190–1° (187–8°). Sol. hot H_2O, hot EtOH.
4-N-*Phenyl*: needles from AcOEt. M.p. 171°.

Bamberger, *Ber.*, 1902, **35**, 1428.
Pinner, *Ber.*, 1888, **21**, 1222.

2-*m*-Tolylsemicarbazide

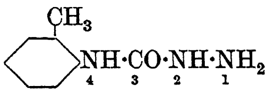


$C_8H_{11}ON_3$ MW, 165

Needles. M.p. 88°. At 140° ⟶ 1-*m*-tolylsemicarbazide.
4-N-*Me*: m.p. 119–20°.
4-N-*Phenyl*: needles from EtOH–Et_2O. M.p. 112°.

Bayer, D.R.P., 163,035, (*Chem. Zentr.*, 1905, II, 1298).

4-*o*-Tolylsemicarbazide



$C_8H_{11}ON_3$ MW, 165

Cryst. from H_2O or EtOH.Aq. M.p. 142–3° decomp.
B,HCl: cryst. from dil. HCl. M.p. 184–6° decomp.

Lei, Sah, Shih, *J. Chinese Chem. Soc.*, 1935, **3**, 246.
Borsche, *Ber.*, 1905, **38**, 835.

4-*m*-Tolylsemicarbazide.
Cryst. from amyl alcohol. M.p. 108°. Forms complexes with $CdCl_2$ and $Ni(NO_3)_2$.

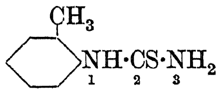
Sah, Wang, Kao, *J. Chinese Chem. Soc.*, 1936, **4**, 187.
Smith, *J. Chem. Soc.*, 1937, 1354.

4-*p*-Tolylsemicarbazide.
Needles from EtOH. M.p. 259–60°. Spar. sol. hot H_2O.
B,HCl: needles from dil. HCl. M.p. 242°.

Sah, Lei, *J. Chinese Chem. Soc.*, 1934, **2**, 167.

Tolyl styryl Ketone.
See 2-, 3-, *and* 4-Methylchalkone.

o-Tolylthiourea



$C_8H_{10}N_2S$ MW, 166

Cryst. from H_2O. M.p. 162° (158°). Sol. EtOH, boiling H_2O. Spar. sol. Et_2O.

1-N-*Me* : *see unsym.*-Methyl-*o*-tolylthiourea.

3-N-*Me* : *see sym.*-Methyl-*o*-tolylthiourea.

3-N-*Et* : *sym.*-ethyl-*o*-tolylthiourea. Prisms from EtOH. M.p. 83–4°.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*o*-tolylthiourea.

Dyson, Hunter, *J. Soc. Chem. Ind.*, 1926, **45**, 81T.
Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 371.
Staats, *Ber.*, 1880, **13**, 136.
Orozdov, *J. Gen. Chem. U.S.S.R.*, 1937, 7, 185.

m-Tolylthiourea.

Prisms from EtOH. M.p. 110–11°. Sol. EtOH, Et_2O. Mod. sol. hot H_2O.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*m*-tolylthiourea.

3-N-*Propionyl* : prisms from EtOH. M.p. 86–7°.

Dyson, Hunter, *J. Soc. Chem. Ind.*, 1926, **45**, 81T.
Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 366, 377.

p-Tolylthiourea.

Plates from EtOH. M.p. 188° (182°). Mod. sol. hot EtOH. Spar. sol. cold H_2O.

3-N-*Me* : *see sym.*-Methyl-*p*-tolylthiourea.

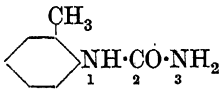
3-N-*Et* : *sym.*-ethyl-*p*-tolylthiourea. Prisms from EtOH. M.p. 95–6°.

3-N *Isoamyl* : *sym.*-isoamyl-*p*-tolylthiourea. Needles from EtOH. M.p. 217° decomp.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*p*-tolylthiourea.

I.G., F.P., 762,310, (*Chem. Zentr.*, 1934, II, 1992).
Staats, *Ber.*, 1880, **13**, 136.
Clermont, Wehrlin, *Bull. soc. chim.*, 1876, **26**, 126.

o-Tolylurea



$C_8H_{10}ON_2$ MW, 150

Leaflets from EtOH. M.p. 190–1° (182°). Sol. $EtOH_2$ Et_2O. Insol. cold H_2O.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*o*-tolylurea.

3-N-o-*Nitrophenyl* : cryst. from EtOH. M.p. 189°.

3-N-*Acetyl* : needles from EtOH. M.p. 168–9°.

3-N-*Isobutyryl* : needles from EtOH. M.p. 134–5°.

3-N-*Palmityl* : needles from EtOH. M.p. 98°.

3-N-*Stearyl* : m.p. 94–5°.

3-N-*Benzoyl* : needles from AcOH. M.p. 210°.

Lei, Sah, Shih, *J. Chinese Chem. Soc.*, 1935, **3**, 246.
v. Braun, *Ber.*, 1908, **41**, 2152.
Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 273.

m-Tolylurea.

Leaflets from H_2O. M.p. 142°.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*m*-tolylurea.

1-N-*Nitroso* : yellow needles from Et_2O–pet. ether. M.p. 80°.

Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 275.
Pierron, *Bull. soc. chim.*, 1906, **35**, 1200.

p-Tolylurea.

Needles from H_2O. M.p. 182–3° (172°). Sol. EtOH.

1-N-*Me* : *see unsym.*-Methyl-*p*-tolylurea.

3-N-*Phenyl* : *see N*-Phenyl-*N'*-*p*-tolylurea.

3-N-*Acetyl* : needles from EtOH. M.p. 199–200°.

3-N-*Isobutyryl* : needles from EtOH. M.p. 138–9°.

3-N-*Palmityl* : m.p. 89–90°.

3-N-*Benzoyl* : needles from EtOH. M.p. 222–3°.

1-N-*Nitroso* : yellow cryst. from Et_2O–pet. ether. M.p. 83°.

Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 275.
Sah, *Sci. reps. Natl. Tsinghua Univ.*, 1934, **2**, 227.
Thate, *Rec. trav. chim.*, 1929, **48**, 116.

p-Tolylurethane.

See under p-Tolylcarbamic Acid.

p-Tolyl p-xylyl Ketone.

See 4 : 4'-Dimethyldeoxybenzoin.

Tolysin.

See under 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

Tomatidine

$C_{27}H_{45(43)}O_2N$ MW, 415(413)

Cryst. M.p. 210–11°. $[\alpha]_D^{20}$ +·5° in MeOH. Forms a digitonide.

B,HCl : cryst. from EtOH. M.p. 275–80° decomp. $[\alpha]_D^{25}$ − 9° in MeOH.

N : O-*Diacetyl deriv.* : m.p. 193–4°.

N : O : O'-*Triacetyl deriv.* : m.p. 105–7°.

Fontaine *et al.*, *Arch. Biochem.*, 1948, **18**, 467.
Kuhn, Löw, *Ber.*, 1948, **81**, 552.
Kuhn, Löw, Gauhe, *Ber.*, 1950, **83**, 448.
Fontaine, Ard, Ma, *J. Am. Chem. Soc.*, 1951, **73**, 878.
Sato, Katz, Mosettig, *J. Am. Chem. Soc.*, 1951, **73**, 880.
Brink, Folkers, *J. Am. Chem. Soc.*, 1951, **73**, 4018.

Tomatine (*Lycopersicin*)

$C_{50}H_{83(81)}O_{21}N$ MW, 1033(1031)

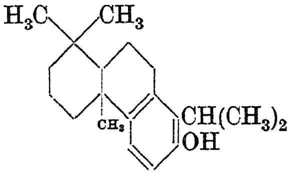
Antifungal glycoside isolated from the dry leaves of the Red Currant tomato, *L. pimpinellifolium.* Also isolated from species of wild tomato plants. Needles from MeOH. M.p. about 270°. $[\alpha]_D^{20}$ $-30°$ in Py. Hyd.$\longrightarrow$ tomatidine + 1 mol. xylose, 1 mol. galactose and 2 mols. glucose.

Fontaine *et al.*, *Arch. Biochem.*, 1948, **18**, 467.
Kuhn, Löw, *Ber.*, 1948, **81**, 552.
Kuhn, Löw, Gauhe, *Ber.*, 1950, **83**, 448.
Ma, Fontaine, *Arch. Biochem.*, 1950, **27**, 461.
Fontaine, Ard, Ma, *J. Am. Chem. Soc.*, 1951, **73**, 878.

Torulin.

See Aneurin.

Totarol



Suggested structure

$C_{20}H_{30}O$ MW, 286

Diterpene phenol occurring in *Podocarpus totara.* Needles from ligroin. M.p. 132°. $[\alpha]_D^{20}$ $+41{\cdot}3°$ in EtOH. Heat with Se$\longrightarrow$ 7-hydroxy-1-methylphenanthrene and 1-methylphenanthrene.

Formyl: hexagonal prisms from EtOH. M.p. 125·5°.

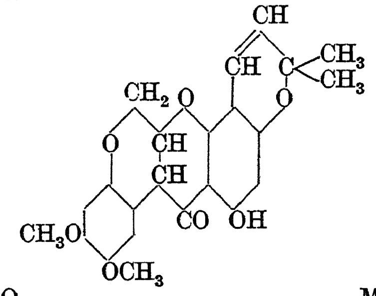
Acetyl: rectangular plates from EtOH. M.p. 121·5°. $[\alpha]_D^{18}$ $+44{\cdot}6°$ in Et_2O.

Hydrogen phthaloyl: prisms from EtOH. M.p. 161–3°.

Me ether: cryst. from $CHCl_3$–EtOH. M.p. 92–92·5°. $[\alpha]_D^{18}$ $+41{\cdot}95°$ in Et_2O.

Short, Stromberg, *J. Chem. Soc.*, 1937, 516.
Short, Wang, *J. Chem. Soc.*, 1950, 991.
Simonsen, Barton, *The Terpenes*, Vol. III, 358 (Cambridge University Press, 1952).

α-Toxicarol



$C_{23}H_{22}O_7$ MW, 410

l-.

Obtained from derris root. Greenish-yellow plates or needles from AcOEt–EtOH. M.p. 125—7° (101–2°). $[\alpha]_D^{20}$ $-66°$ in C_6H_6, $+58°$ in Me_2CO. Fish poison. Properties closely related to those of rotenone.

Monoacetyl deriv.: m.p. 158°.

dl-.

M.p. 219–23° (231–2°).

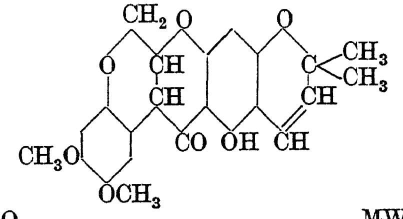
Monoacetyl deriv.: cryst. from Me_2CO–pet. ether. M.p. 182·5°.

Diacetyl deriv.: cryst. from AcOH. M.p. 220–3°.

Benzoyl deriv.: needles from C_6H_6–$CHCl_3$. M.p. 202°.

Heyes, Robertson, *J. Chem. Soc.*, 1935, 681.
Butenandt, Hilgetag, *Ann.*, 1933, **506**, 169.
Clark, *J. Am. Chem. Soc.*, 1930, **52**, 246.
George, Robertson, *J. Chem. Soc.*, 1937, 1535.
Harper, *J. Chem. Soc.*, 1939, 812.
Cahn, Phipers, Boam, *J. Chem. Soc.*, 1938, 513, 734.

β-Toxicarol



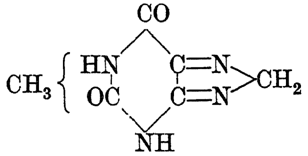
$C_{23}H_{22}O_7$ MW, 410

dl-.

Pale yellow plates from EtOH. M.p. 169–70°.

Cahn, Phipers, Boam, *J. Chem. Soc.*, 1938, 513.

Toxoflavin



Suggested structure

$C_6H_6O_2N_4$ MW, 166

Needles. M.p. 172°. Sol. H_2O, EtOH. Spar. sol. AcOEt, $CHCl_3$.

Veen, Mertens, *Rec. trav. chim.*, 1934, **53**, 398.
Veen, Baars, *Rec. trav. chim.*, 1938, **57**, 248.

T. P. N.

See Coenzyme II.

Trasentin (*Hydrochloride of 2-diethylaminoethyl diphenylacetate*)

$(C_6H_5)_2CH{\cdot}COO{\cdot}CH_2{\cdot}CH_2N(C_2H_5)_2,HCl$

$C_{20}H_{26}O_2NCl$ MW, 347·5

Cryst. M.p. 113–14°. Sol. H_2O. Parasympathetic antispasmodic.

Ciba, Swiss Ps., 190,541, 192,070, 192,697, (*Chem. Abstracts*, 1938, **32**, 589, 4174, 4284).

Traumatic Acid (1-*Decylene*-1 : 10-*dicarboxylic acid*)

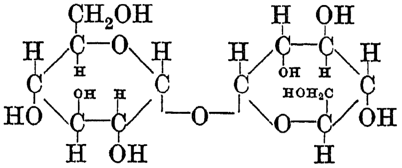
$HOOC{\cdot}CH_2{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}COOH$

$C_{12}H_{20}O_4$ MW, 228

Plant wound hormone isolated from string bean pods. Cryst. from EtOH or Me_2CO. M.p. 165–6°.

English, Benner, Haagen-Smit, *J. Am. Chem. Soc.*, 1939, **61**, 3434.

α : α-**Trehalose** (*Mycose*)



$C_{12}H_{22}O_{11}$ MW, 342

Occurs in fungi, moulds, ergot, algæ, yeast, etc. Prisms $+ 2H_2O$ from EtOH. M.p. hydrate 97°, anhyd. 210° (203°). Sweet taste. $[\alpha]_D^{20}$ hydrate $+ 178{\cdot}3°$ in H_2O, $[\alpha]_D^{20}$ anhyd. $+ 197{\cdot}0°$ in H_2O. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. Does not reduce Fehling's. Fermentable by most yeasts. Does not form an osazone. Hyd. by dil. acids ⟶ glucose. HNO_3 ⟶ oxalic acid.

Hexa-acetyl : m.p. 93–6°. $[\alpha]_D^{19}$ $+ 158{\cdot}3°$ in $CHCl_3$.

Octa-acetyl : cryst. from EtOH. M.p. 97–8° (97°, 96–8°, 80°, 70–5°, 100–2° after drying in vacuo). $[\alpha]_D^{20}$ $+ 162{\cdot}3°$ in $CHCl_3$.

Octa-nitrate : laminæ from EtOH. M.p. 124°. Decomp. at 136°. $[\alpha]_D^{18}$ $+ 173{\cdot}8°$ in AcOH.

Octa-Me ether : yellow liq. B.p. 170°/0·03 mm. $[\alpha]_D^{20}$ $+ 199{\cdot}8°$ ($+ 186{\cdot}1°$) in C_6H_6.

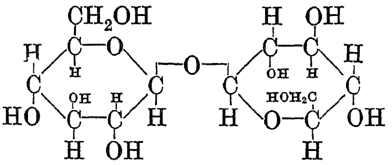
6-*Mesyl* : hygroscopic powder. $[\alpha]_D^{20}$ $+ 139°$ in H_2O.

6-*Mesyl-hepta-acetyl* : m.p. 84°. $[\alpha]_D^{20}$ $+ 139°$ in AcOEt.

Octa-mesyl : decomp. at 250°. $[\alpha]_D^{20}$ $+ 105{\cdot}5°$ in AcOEt.

Schlubach, Maurer, *Ber.*, 1925, **58**, 1183.
Berthelot, *Ann. chim. phys.*, 1859, **55**, 272, 291.
v. Lippmann, *Ber.*, 1912, **45**, 3431.
Will, Lenze, *Ber.*, 1898, **31**, 85.
Pangborn, Anderson, *J. Biol. Chem.*, 1933, **101**, 105.
Bredereck, *Ber.*, 1930, **63**, 959.
Harding, *Sugar*, 1923, **25**, 476, (*Chem. Abstracts*, 1924, **18**, 78).
Myrbäck, Oerteblad, *Biochem. Z.*, 1936, **288**, 329.

β : β-**Trehalose**



$C_{12}H_{22}O_{11}$ MW, 342

$[\alpha]_D$ $- 39{\cdot}4°$.

Octa-Me ether : b.p. 160°/0·015 mm. $[\alpha]_D^{20}$ $+ 82{\cdot}8°$ in C_6H_6.

Schlubach, Mauer, *Ber.*, 1925, **58**, 1179.
Irvine *et al.*, *J. Am. Chem. Soc.*, 1929, **51**, 3609.

Triacetic Acid (2 : 4-*Diketocaproic acid*)

$CH_3{\cdot}CO{\cdot}CH_2{\cdot}CO{\cdot}CH_2{\cdot}COOH$

$C_6H_8O_4$ MW, 144

Yellow plates from Et_2O–pet. ether. M.p. 29–31°. pK 3·3. Sol. H_2O, $CHCl_3$, Et_2O, dioxan, AcOEt. Insol. pet. ether. Unstable in H_2O. Aq.$FeCl_3$ ⟶ red col. NaOH.Aq. ⟶ AcOH + CO_2 + Me_2CO.

Lactone : long needles. M.p. 186–7°. *Oxime* : needles from EtOH.Aq. M.p. 230–1°.

2 : 4-*Dinitrophenylhydrazone* : (i) yellow cryst. from AcOEt–pet. ether. M.p. 189–91° decomp. (ii) Yellow needles from AcOEt–pet. ether. M.p. 149–50°.

Collie, *J. Chem. Soc.*, 1891, **59**, 607.
Witter, Stotz, *J. Biol. Chem.*, 1948, **176**, 485.

Triacetin (*Triacetylglycerol, glycerol triacetate*)

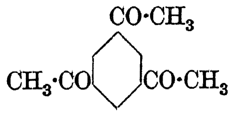
$CH_2{\cdot}O{\cdot}CO{\cdot}CH_3$
$CH{\cdot}O{\cdot}CO{\cdot}CH_3$
$CH_2{\cdot}O{\cdot}CO{\cdot}CH_3$

$C_9H_{14}O_6$ MW, 218

Colourless liq. B.p. 258–60°, 172°/40 mm. Sol. EtOH, Et_2O, C_6H_6, $CHCl_3$. Sol. H_2O to 70% at 15°. Prac. insol. CS_2, ligroin. D_4^{25} 1·1562.

Perkin, Simonsen, *J. Chem. Soc.*, 1905, **87**, 858.
Böttinger, *Ann.*, 1891, **263**, 359.

1 : 3 : 5-Triacetobenzene (1 : 3 : 5-*Triacetylbenzene*)

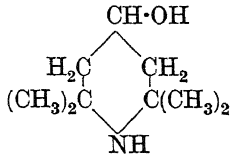


$C_{12}H_{12}O_3$ MW, 204

Needles from EtOH or AcOH. M.p. 163°. Sol. AcOH. Spar. sol. H_2O, EtOH, Et_2O. HNO_3 ⟶ trimesic acid.

Viguier, *Compt. rend.*, 1911, **153**, 1232.
Claisen, Stylos, *Ber.*, 1888, **21**, 1145.
Frank, Varland, *Organic Syntheses*, 1947, XXVII, 91.

Triacetonalkamine (*4-Hydroxy-2 : 2 : 6 : 6-tetramethylpiperidine*)



$C_9H_{19}ON$ MW, 157

Plates from Et_2O, hydrated cryst. from H_2O. M.p. 129°. Very sol. H_2O. Spar. sol. Et_2O. Sublimes.

N-*Me*: $C_{10}H_{21}ON$. MW, 171. Hydrated plates from H_2O. M.p. anhyd. 74°. B.p. 125°/12 mm. Very sol. warm H_2O.

O : N-*Di-Me*: $C_{11}H_{23}ON$. MW, 185. Oil. B.p. 217–21°. Spar. sol. H_2O.

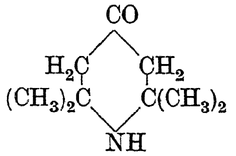
O-*Benzoyl*: needles from EtOH.Aq. M.p. 97–8°. *B,HCl*: m.p. 240°.

O : N-*Dibenzoyl*: cryst. $+ 1H_2O$ from EtOH.Aq. M.p. 200°.

N-*Nitroso*: pale yellow needles from C_6H_6–pet. ether. M.p. 93°.

Orthner, *Ann.*, 1927, **456**, 252.
Harries, *Ann.*, 1918, **417**, 121.
Clarke, Francis, *Ber.*, 1912, **45**, 2060.

Triacetonamine (*4-Keto-2 : 2 : 6 : 6-tetramethylpiperidine, 2 : 2 : 6 : 6-tetramethyl-γ-piperidone*)



$C_9H_{17}ON$ MW, 155

Plates $+ 1H_2O$ from Et_2O, m.p. 58°. Anhyd. needles from dry Et_2O, m.p. 34·9°. B.p. 205°. Sol. H_2O, EtOH, Et_2O. Red. ⟶ triacetonalkamine. Forms unstable Na deriv.

B_2,H_2CrO_4: yellow prisms. Decomp. at 105°.

Oxime: prisms from EtOH. M.p. 153°.

Semicarbazone: cryst. from EtOH. M.p. 219–20°.

N-*Me*: 1 : 2 : 2 : 6 : 6-pentamethyl-γ-piperidone. $C_{10}H_{19}ON$. MW, 169. Yellow liq. with unpleasant odour. B.p. 200° decomp., 122°/23 mm. *B,HI*: m.p. 172°.

N-*Et*: $C_{11}H_{21}ON$. MW, 183. Liq. *B_2,H_2PtCl_6*: orange prisms from H_2O. M.p. 157–8° decomp.

N-*Allyl*: $C_{12}H_{21}ON$. MW, 195. Liq. *B_2,H_2PtCl_6*: orange-yellow plates or prisms. M.p. 148°.

N-*Benzyl*: $C_{16}H_{23}ON$. MW, 245. *B,HCl*: plates or prisms from EtOH–Et_2O. M.p. 137–8°. *B_2,H_2PtCl_6*: orange-red cryst. M.p. 147–8° decomp.

N-*Bromo*: $C_9H_{16}ONBr$. MW, 234. Cryst. from pet. ether. M.p. 44°. Spar. sol. H_2O.

N-*Nitroso*: needles from H_2O or EtOH.Aq. M.p. 72–3°. Very sol. EtOH, Et_2O. Volatile in steam. Sublimes.

N-*OH*: triacetone-hydroxylamine. $C_9H_{17}O_2N$. MW, 171. Cryst. from pet. ether. M.p. 50–1°. Sol. dil. alkalis and acids. Reduces $NH_3.AgNO_3$. *B,HI*: m.p. 180°. *Oxime*: cryst. from pet. ether. M.p. 126–7°.

Francis, *J. Chem. Soc.*, 1927, 2897.
Clarke, Francis, *Ber.*, 1912, **45**, 2064.
Harries, *Ann.*, 1918, **417**, 171.
Harries, Lehmann, *Ber.*, 1897, **30**, 232, 2736.

Triacetone-hydroxylamine.
See under Triacetonamine.

Triacetylene (*Hexatri-yne*)

H·C≡C·C≡C·C≡CH

C_6H_2 MW, 74

White microcrystalline solid, stable in light *in vacuo* for several minutes at liquid-air temperature. Slowly becomes red at about − 50°. Melts below 0°. Can detonate with remarkable violence at room temperature.

Armitage, Cook, Jones, Whiting, *J. Chem. Soc.*, 1952, 2010.
Hunsmann, *Ber.*, 1950, **83**, 213.

Triacontane

$CH_3·[CH_2]_{28}·CH_3$

$C_{30}H_{62}$ MW, 422

Constituent of many mineral oils, of the wax coating of apple skins and of numerous flowers, *Arnica montana, Linaria vulgaris*, etc. Plates from C_6H_6. M.p. 66°. B.p. 235°/1 mm., 202°/0·05 mm. D^{90} 0·7676. n_D^{90} 1·4266. Very sol. hot C_6H_6. Spar. sol. hot EtOH.

Landa, *Chem. Abstracts*, 1929, **23**, 4667.
Gascard, *Ann. chim.*, 1921, **15**, 332.
Magee, *Rec. trav. chim.*, 1948, **67**, 197.

***n*-Triacontanic Acid** (*Melissic acid*)

$CH_3·[CH_2]_{28}·COOH$

$C_{30}H_{60}O_2$ MW, 452

Occurs in candelilla wax. Colourless plates from Me_2CO. M.p. 93·5–94°. Spar. sol. Et_2O, Me_2CO.

Me ester: $C_{31}H_{62}O_2$. MW, 466. Colourless plates from pet. ether. M.p. 71·5°.

Et ester: $C_{32}H_{64}O_2$. MW, 480. Colourless plates from EtOH. M.p. 70·5°.

Robinson, *J. Chem. Soc.*, 1934, 1544.

***n*-Triacontanol** (*1-Hydroxytriacontane, melissyl alcohol, myricyl alcohol*)

$CH_3·[CH_2]_{28}·CH_2OH$

$C_{30}H_{62}O$ MW, 438

Present in *Arbutus unedo* and candelilla wax, and in beeswax as palmitate. Plates from C_6H_6. M.p. 86·5°.

Acetyl: plates from pet. ether. M.p. 69°.

Robinson, *J. Chem. Soc.*, 1934, 1545.

Triallylamine

$(CH_2:CH \cdot CH_2)_3N$

$C_9H_{15}N$ MW, 137

Liq. with unpleasant odour. B.p. 155–6°. D^{14} 0·8094.

Grosheintz, *Bull. soc. chim.*, 1879, **31**, 391.

Triallyl phosphate.

See under Phosphoric Acid.

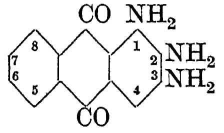
Triallyltricyanamide (*Sinamin*, *allylcyanamide*)

$C_{12}H_{18}N_6$ MW, 246

Cryst. + $\frac{1}{2}H_2O$ from H_2O. M.p. 100° with loss of H_2O. Strongly alkaline.

Will, *Ann.*, 1844, **52**, 15.

1 : 2 : 3-Triaminoanthraquinone



$C_{14}H_{11}O_2N_3$ MW, 253

Black needles from $PhNO_2$. M.p. 325° decomp. Sol. conc. $H_2SO_4 \longrightarrow$ red sol.

Scholl, Eberle, Tritsch, *Monatsh.*, 1911, **32**, 1044.

Scholl, Schneider, Eberle, *Ber.*, 1904, **37**, 4438.

1 : 2 : 4-Triaminoanthraquinone.

Bluish-red powder. Does not melt below 300°.

1 : 2 : 4 - N - *Triphenyl* : 1 : 2 : 4 - trianilino-anthraquinone. $C_{32}H_{23}O_2N_3$. MW, 481. Black needles from $PhNO_2$ or Py. Spar. sol. EtOH. Conc. $H_2SO_4 \longrightarrow$ yellow sol. $\longrightarrow$ red on warming.

Terres, *Monatsh.*, 1921, **41**, 608.

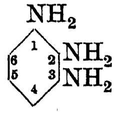
Bayer, D.R.P., 151,511, (*Chem. Zentr.*, 1904, I, 1507).

1 : 4 : 5-Triaminoanthraquinone.

M.p. 256–7°.

S.C.I., F.P. 811,479, (*Chem. Abstracts*, 1938, **32**, 591).

1 : 2 : 3-Triaminobenzene



$C_6H_9N_3$ MW, 123

Cryst. M.p. up to 103°. B.p. 336°. Sol. H_2O, EtOH, Et_2O. Reacts strongly alkaline. $FeCl_3 \longrightarrow$ violet $\longrightarrow$ brown ppt. Sol. in conc. H_2SO_4 + trace $HNO_3 \longrightarrow$ dark blue col. Reduces $NH_3.AgNO_3$. Boiling AcOH $\longrightarrow$ 4-acetylamino-2-methylbenziminazole.

B,2HCl : m.p. above 300°.

2-N-*Phenyl* : *see* 2 : 6-Diaminodiphenylamine.

Dipicrate : yellow cryst. Decomp. at 183°.

Salkowski, *Ann.*, 1872, **163**, 23.

Platt, Sharp, *J. Chem. Soc.*, 1948, 2129.

1 : 2 : 4-Triaminobenzene.

Plates from $CHCl_3$. M.p. 95–8°. B.p. about 340°. Sol. H_2O, EtOH. Mod. sol. $CHCl_3$. Spar. sol. Et_2O. Turns brown in air. $FeCl_3 \longrightarrow$ red col.

1-N-*Di-Me* : 2 : 4-diamino-*N*-dimethylaniline. $C_8H_{13}N_3$. MW, 151. Needles from ligroin. M.p. 44°. B.p. 218–19°/90 mm., 178°/22 mm. Sol. H_2O. Turns dark blue in air. Aq. sol. + ox. agents $\longrightarrow$ red sol. *B,2HCl* : cryst. M.p. 225°. *B,2HBr* : m.p. 207° decomp. *B,2HI* : plates + $\frac{1}{2}$EtOH from EtOH. M.p. 190° decomp. 2 : 4-N-*Diacetyl* : cryst. + $1\frac{1}{2}H_2O$ from H_2O, m.p. 82°; cryst. from C_6H_6 or AcOEt, m.p. 153°.

2 : 4-N-*Tetra-Me* : $C_{10}H_{17}N_3$. MW, 179. Liq. B.p. 209·4°/112 mm., 180·5°/45 mm. D_4^{22} 1·0203. Has caustic action. *B,2HCl* : cryst. powder. M.p. 164°. *B,2HBr* : m.p. 179°. *B,2HI* : prisms. M.p. 175° decomp. *Picrate* : m.p. 169° decomp. 1-N-*Acetyl* : plates from ligroin. M.p. 85°. 1-N-*Benzenesulphonyl* : prisms from ligrion. M.p. 84°.

1 : 2 : 4-N-*Hexa-Me* : $C_{12}H_{21}N_3$. MW, 207. Liq. B.p. 210°/136 mm., 184°/40 mm. *Trimethiodide* : needles + 2MeOH from MeOH. M.p. 164·5° decomp.

1-N-*Phenyl* : *see* 2 : 4-Diaminodiphenylamine.

2 : 4 - N - *Diphenyl* : 4-amino-3-anilinodiphenylamine. $C_{18}H_{17}N_3$. MW, 275. Needles from C_6H_6–ligroin. M.p. 107°. Spar. sol. H_2O, ligroin. Sol. Et_2O, C_6H_6.

1-N-*Acetyl* : 2 : 4-diaminoacetanilide. Prisms. M.p. 158–9°.

1 : 4-N-*Diacetyl* : cryst. M.p. 231–2°.

1 : 2 : 4-N-*Tribenzoyl* : needles from AcOH. M.p. 260°.

Hinsberg, *Ber.*, 1886, **19**, 1253.

Wurster, Sendtner, *Ber.*, 1879, **12**, 1806.

Ragno, Bruno, *Gazz. chim. ital.*, 1946, **76**, 485.

1 : 3 : 5-Triaminobenzene.

White needles. M.p. 129° (from $CHCl_3$), 118° (from C_6H_6). Stable in the dark. Darkens slowly in the light. Forms a monohydrate, m.p. 84–5°.

1 : 3 : 5-N-*Triphenyl* : 1 : 3 : 5-trianilinobenzene. $C_{24}H_{21}N_3$. MW, 351. Needles from EtOH. M.p. 193°. Sol. Et_2O. Spar. sol. cold EtOH, C_6H_6. Conc. $H_2SO_4 \longrightarrow$ violet-red col. on warming. *B,HCl* : yellow powder. M.p. below 100°. B_2,H_2PtCl_6 : yellow ppt. M.p. 251° decomp. 1 : 3 : 5-N-*Trinitroso* : brown needles from EtOH. M.p. 264–5°. 1 : 3 : 5-N-*Triacetyl* : needles from EtOH. M.p. 172–3°.

1 : 3 : 5-N-*Tri*-p-*tolyl* : $C_{27}H_{27}N_3$. MW, 393. Needles from EtOH. M.p. 186–7°. Spar. sol. cold EtOH. Conc. $H_2SO_4 \longrightarrow$ bluish-green

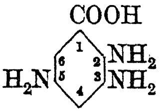
on warming. 1 : 3 : 5-N-*Trinitroso* : needles from EtOH. M.p. 233–4°. 1 : 3 : 5-N-*Triacetyl* : needles from EtOH. M.p. 192–3°. 1 : 3 : 5-N-*Tribenzoyl* : prisms from EtOH. M.p. 281–2°.

1 : 3 : 5-N-*Triacetyl* : plates from EtOH. M.p. 208°.

1 : 3 : 5-N-*Tribenzoyl* : needles from EtOH–C_6H_6. Does not melt below 350°.

Minunni, *Gazz. chim. ital.*, 1890, **20**, 322.
Hepp, *Ann.*, 1882, **215**, 348.
Kreuger, U.S.P. 2,461,498, (*Chem. Abstracts*, 1949, **43**, 3456).
McLean, Tetlow, Munro, B.P. 589,716, (*Chem. Abstracts*, 1948, **42**, 217).

2 : 3 : 5-Triaminobenzoic Acid



$C_7H_9O_2N_3$ MW, 167

Cryst. from H_2O. Chars on heating evolving NH_3. Very sol. hot H_2O. Spar. sol. hot EtOH. Insol. Et_2O. Aq. sol. rapidly becomes red. $FeCl_3 \longrightarrow$ brown ppt.

2-N-*Phenyl* : 3 : 5-diamino-2-anilinobenzoic acid, 4 : 6-diaminodiphenylamine-2-carboxylic acid. $C_{13}H_{13}O_2N_3$. MW, 243. Cryst. M.p. 237–8° decomp. Very sol. EtOH, AcOH. Spar. sol. Et_2O, C_6H_6, hot H_2O.

Cohn, Schifferes, *Chem. Zentr.*, 1902, I, 1293.
Griess, *Ber.*, 1882, **15**, 2199.

2 : 4 : 6-Triaminobenzoic Acid.

$HNO_3 \longrightarrow$ intense yellow sol., violet on neutralisation. Hydrochloride + boiling $H_2O \longrightarrow$ phloroglucinol.

Hydrochloride : prisms from H_2O.

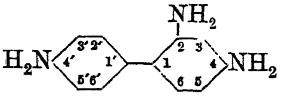
Cassella, D.R.P., 102,358, (*Chem. Zentr.*, 1899, I, 1263).
Phillips, Lowry, *Transactions of the Electrochemical Society*, 1937, **71**, 10.
Lewis, Brown, *Transactions of the Electrochemical Society*, 1943, **84**, 9.

3 : 4 : 5-Triaminobenzoic Acid.

Needles + $\frac{1}{2}H_2O$ from hot H_2O. Loses H_2O at 100°. Sol. hot H_2O. Spar. sol. cold H_2O. Insol. hot EtOH. Reacts acid. Dist. $\longrightarrow$ 1 : 2 : 3-triaminobenzene. Sol. in conc. H_2SO_4 + trace $HNO_3 \longrightarrow$ bluish-green $\longrightarrow$ dark blue col.

Salkowski, *Ann.*, 1872, **163**, 12.

2 : 4 : 4′-Triaminodiphenyl (2-*Aminobenzidine*)



$C_{12}H_{13}N_3$ MW, 199

Needles. M.p. 134°.

4 : 4′-N-*Di*-p-*toluenesulphonyl* : m.p. 198°.

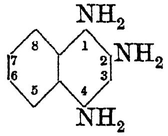
Tauber, *Ber.*, 1890, **23**, 798.

2 : 5 : 4′-Triaminodiphenyl.

5-N-*Di-Me* : $C_{14}H_{17}N_3$. MW, 227. Cryst. M.p. 87–9°. Sol. EtOH, dil. min. acids. Spar. sol. H_2O, ligroin. $FeCl_3 \longrightarrow$ violet $\longrightarrow$ blue col. *Picrate* : yellow prisms. M.p. 127°. 2 : 4′-N-*Diacetyl* : needles from EtOH.Aq. M.p. 233°.

Jacobson, Kunz, *Ann.*, 1898, **303**, 354.

1 : 2 : 4-Triaminonaphthalene



$C_{10}H_{11}N_3$ MW, 173

1-N-*Phenyl* : 2 : 4-diamino-1-anilinonaphthalene, 2 : 4-diamino-1-*N*-phenylnaphthylamine. $C_{16}H_{15}N_3$. MW, 249. Needles from C_6H_6. M.p. 190°. Sol. AcOH, C_6H_6. Very spar. sol. ligroin, Et_2O.

1 : 2 : 4-N-*Triphenyl* : 1 : 2 : 4-trianilinonaphthalene. $C_{28}H_{23}N_3$. MW, 401. Needles from EtOH or C_6H_6–ligroin. M.p. 148°.

1-N-p-*Tolyl* : 2 : 4-diamino-1-*N*-*p*-tolylnaphthylamine. $C_{17}H_{17}N_3$. MW, 263. Needles from C_6H_6. M.p. 176–7°. Very readily oxidised. Resinifies in air.

1 : 2 : 4-N-*Tri*-p-*tolyl* : $C_{31}H_{29}N_3$. MW, 443. Needles from EtOH. M.p. 159–60°.

1-N-*Acetyl* : 2 : 4-diamino-1-acetnaphthalide. Brown needles. M.p. 189°.

1 : 2 : 4-N-*Triacetyl* : white cryst. M.p. 301°.

1-N-*Benzenesulphonyl* : needles from toluene–pet. ether. M.p. 195–7°.

Panizzon-Favre, *Gazz. chim. ital.*, 1924, **54**, 826.
Ullmann, Bruck, *Ber.*, 1908, **41**, 3937.
Fischer, Hepp, *Ann.*, 1890, **256**, 250.

1 : 2 : 5-Triaminonaphthalene.

B,HCl : cryst. Turns brown in H_2O.

1 : 2 : 5-N-*Tribenzoyl* : yellow powder. M.p. 268°.

Finzi, *Chem. Abstracts*, 1925, **19**, 2661.

1 : 2 : 6-Triaminonaphthalene.

2-N-*Me* : $C_{11}H_{13}N_3$. MW, 187. 2-N-p-*Toluenesulphonyl* : prisms from C_6H_6–pet. ether. Decomp. at 185°.

1 : 2 : 6-N-*Triacetyl* : needles from AcOH. M.p. 280° decomp.

1 : 2 : 6-N-*Tribenzoyl* : needles from AcOH. M.p. 277°.

2-N-p-*Toluenesulphonyl* : needles from toluene. M.p. 190°.

Loewe, *Ber.*, 1890, **23**, 2544.

1 : 2 : 7-Triaminonaphthalene.

Free base not known.

B,HCl : cyst. from HCl. Sinters at 200°.

Hey, Lawton, *J. Chem. Soc.*, 1940, 384.

1 : 3 : 6-Triaminonaphthalene.

Sol. H_2O. Acid. sol. + nitrite ⟶ deep brown col.

Sulphate : needles. Spar. sol. H_2O.

Kalle, D.R.P., 89,061.

1 : 3 : 7-Triaminonaphthalene.

Sol. H_2O, EtOH. Aq. sol. + HNO_3 ⟶ deep brown col. Salt sols + $FeCl_3$ ⟶ violet col.

Sulphate : cryst. Spar. sol. hot H_2O.

Kalle, D.R.P., 90,905.

1 : 3 : 8-Triaminonaphthalene.

B,3HI : needles.

Aguiar, Lautemann, *Bull. soc. chim.*, 1865, 3, 263.

1 : 2 : 3-Triaminopropane

$H_2N{\cdot}CH_2{\cdot}CH(NH_2){\cdot}CH_2{\cdot}NH_2$

$C_3H_{11}N_3$ MW, 89

Viscous oil. B.p. 190°, 92–3°/9 mm. Sol. H_2O.

B,3HCl,H₂O : plates. M.p. 250°.

B,3HCl,AuCl₃ : yellow prisms. M.p. 210–12°.

B,3HCl,PtCl₄ : yellow needles. M.p. 220° decomp.

B,2HCl,HI : cryst. from H_2O. M.p. 303–4° decomp.

B,3HBr : m.p. 307–10°.

Triacetyl : cryst. from EtOH. M.p. 200–2°.

Tribenzoyl : m.p. 217–18°.

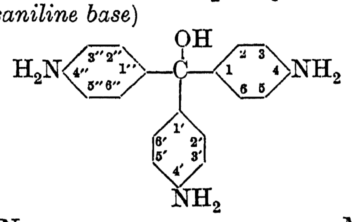
Picrate : yellow needles. Does not melt below 270°.

Pope, Mann, *Compt. rend.*, 1924, **178**, 2085.

Brackebusch, *Ber.*, 1873, **6**, 1290.

4 : 4′ : 4″-Triaminotriphenylcarbinol

(*Pararosaniline base*)



$C_{19}H_{19}ON_3$ MW, 305

Colourless plates. M.p. about 205°. Turns red in air. Very spar sol. H_2O. Sol. EtOH. Insol. Et_2O. Heat of comb. C_v 2481·0 Cal., C_p 2483·5 Cal. Red. ⟶ paraleucaniline. H_2O at 270° ⟶ 4 : 4′-dihydroxybenzophenone. KCN ⟶ triaminotriphenylacetonitrile.

Me ether : $C_{20}H_{21}ON_3$. MW, 319. Plates + $1Et_2O$ from Et_2O. m.p. 105°; plates + $1C_6H_6$ from C_6H_6, m.p. 135°.

4 : 4′ : 4″-N-*Triacetyl* : needles from Me_2CO–Et_2O. M.p. 192°.

Wieland, Scheuing, *Ber.*, 1921, **54**, 2527.

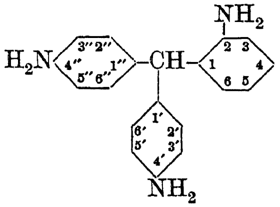
M.L.B., D.R.P., 300,467, (*Chem. Zentr.*, 1917, II, 579).

Zimmermann, Müller, *Ber.*, 1885, **18**, 997.

Fischer, *Ber.*, 1882, **15**, 678; 1880, **13**, 2205.

Fischer, *Ann.*, 1878, **194**, 274.

2 : 4′ : 4″-Triaminotriphenylmethane



$C_{19}H_{19}N_3$ MW, 289

Cryst. from EtOH. M.p. 165°.

Renouf, *Ber.*, 1883, **16**, 1305.

3 : 4′ : 4″-Triaminotriphenylmethane.

Needles + $1C_6H_6$ from C_6H_6, m.p. 145°; cryst. from Et_2O–petrol, m.p. 150°. Sol. EtOH.

Fischer, Ziegler, *Ber.*, 1880, **13**, 672.

4 : 4′ : 4″-Triaminotriphenylmethane

(*Paraleucaniline*).

Plates from H_2O, EtOH or C_6H_6. M.p. 202·5° (208°). Ox. ⟶ pararosaniline base. Diazotise + EtOH ⟶ triphenylmethane.

4 : 4′ : 4″-N-*Triacetyl* : reddish plates from EtOH. M.p. 201°.

B,C₆H₃(NO₂)₃-1 : 3 : 5 : black prisms. M.p. 140°.

Fischer *et al.*, *J. prakt. Chem.*, 1909, **79**, 563; *Ann.*, 1878, **194**, 268.

Triaminotriazine.

See Melamine.

3 : 4 : 5-Triamino-1 : 2 : 4-triazole.

See Guanazine.

Tri-*n*-amylamine

$(CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2)_3N$

$C_{15}H_{33}N$ MW, 227

Liq. B.p. 240–5°, 130°/14 mm., 109°/5 mm. D^{20} 0·7907. n_D 1·43665.

Marvel, Scott, Amstutz, *J. Am. Chem. Soc.*, 1929, **51**, 3640.

Mailhe, *Compt. rend.*, 1918, **166**, 997.

Tri-*active*-amylamine

$(CH_3{\cdot}CH_2{\cdot}\overset{CH_3}{\overset{|}{C}H}{\cdot}CH_2)_3N$

$C_{15}H_{33}N$ MW, 227

Liq. B.p. 230–7°, 94°/4 mm. D^{13} 0·7964. n_D 1·43305.

Plimpton, *J. Chem. Soc.*, 1881, **39**, 335.

Tri-*n*-amyl borate

$(CH_3{\cdot}[CH_2]_3{\cdot}CH_2O)_3B$

$C_{15}H_{33}O_3B$ MW, 272

B.p. 274·5–276·1°. D_4^{27} 0·85224. n_D^{20} 1·4197.

Scattergood, Miller, Gammon, *J. Am. Chem. Soc.*, 1945, **67**, 2150.

Triamyl phosphate.
See under Phosphoric Acid.

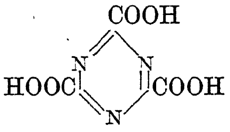
Trianilinoanthraquinone.
See under Triaminoanthraquinone.

Trianilinobenzene.
See under Triaminobenzene.

Trianilinomethane.
See under Orthoformic Acid.

Trianilinonaphthalene.
See under Triaminonaphthalene.

Triazine-tricarboxylic Acid (*Cyanuric tricarboxylic acid, paracyanoformic acid*)



$C_6H_3O_6N_3$ MW, 213

Cryst. powder. M.p. above 250° decomp. Spar. sol. H_2O. Insol. EtOH, Et_2O. Boiling $H_2O \longrightarrow NH_3$ + oxalic acid.

Tri-Me ester: $C_9H_9O_6N_3$. MW, 255. Needles from EtOH. M.p. 154°.

Tri-Et ester: $C_{12}H_{15}O_6N_3$. MW, 297. Needles from EtOH. M.p. 165°.

Tri-isobutyl ester: needles from EtOH. M.p. 158°.

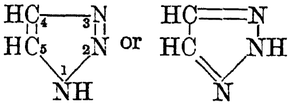
Tri-Me-amide: needles from EtOH or H_2O. M.p. above 250° with decomp.

Trinitrile: C_6N_6. MW, 156. M.p. 119° with sublimation. B.p. 262°.

Weddige, *J. prakt. Chem.*, 1874, **10**, 212.

Triazo-.
See Azido-, *and* individual azides.

1 : 2 : 3-Triazole (*Osotriazole*)



$C_2H_3N_3$ MW, 69

M.p. 23°. B.p. 203°/739 mm. Sol. ord. org. solvents. Insol. ligroin. Hygroscopic.

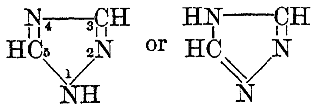
1-N-*Phenyl*: *see* 1-Phenyl-1 : 2 : 3-triazole.

1-N-*Benzyl*: $C_9H_9N_3$. MW, 159. Cryst. M.p. 61°. B.p. 180–3°/16 mm.

1-N-*Benzoyl*: needles from $CHCl_3$ or Et_2O. M.p. 100–2°.

Pechmann, Baver, *Ber.*, 1909, **42**, 673.
Zincke, *Ann.*, 1900, **311**, 317.
Dimroth, *Ber.*, 1902, **35**, 1044.

1 : 2 : 4-Triazole (*Pyrrodiazole*)



$C_2H_3N_3$ MW, 69

Needles from Et_2O or C_6H_6. M.p. 120–1°. B.p. 260°. Sol. H_2O, EtOH. Forms comps. with metals and metallic salts.

B,HCl: plates. M.p. 169°.

Oxalate: cryst. from H_2O. Decomp. at 251°.

B_2,H_2PtCl_6: needles. M.p. 73–5°.

1-*Phenyl*: *see* 1-Phenyl-1 : 2 : 4-triazole.

3-*Phenyl*: *see* 3-Phenyl-1 : 2 : 4-triazole.

Pellizzari, *Gazz. chim. ital.*, 1911, **41**, 20; *Atti accad. Lincei*, 1902, **11**, 20.
Paolini, Baj, *Gazz. chim. ital.*, 1931, **61**, 557.
Bladin, *Ber.*, 1892, **25**, 745.

Tribenzamide

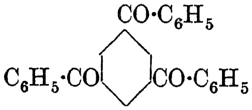
$C_6H_5{\cdot}CO{\cdot}N(CO{\cdot}C_6H_5)_2$

$C_{21}H_{15}O_3N$ MW, 329

Needles from EtOH. M.p. 207–8° (202°). Sublimes. Heat. of comb. 2425·5 Cal. Hyd. by KOH.

Titherley, *J. Chem. Soc.*, 1904, **85**, 1187.
Wheeler, Walden, Metcalf, *J. Am. Chem. Soc.*, 1898, **20**, 73.

1 : 3 : 5-Tribenzoylbenzene



$C_{27}H_{18}O_3$ MW, 390

Needles from EtOH. M.p. 118–19°.

Trioxime: m.p. 198°.

Monophenylhydrazone: m.p. 84°.

Claisen, *Ann.*, 1894, **281**, 307.
Croxall, Van Hook, *J. Am. Chem. Soc.*, 1949, **71**, 2422.

Tribenzoylmethane

$C_6H_5{\cdot}CO{\cdot}CH{<}^{CO\cdot C_6H_5}_{CO\cdot C_6H_5}$

or

$C_6H_5{\cdot}C(OH){:}C{<}^{CO\cdot C_6H_5}_{CO\cdot C_6H_5}$

$C_{22}H_{16}O_3$ MW, 328

α- or Enol form:

Cryst. M.p. (in Jena glass) 155°, (in soft glass) 240–5°. More sol. than keto-form. $FeCl_3 \longrightarrow$ red col. EtOH or $CH_3COCl \longrightarrow$ keto-form.

β- or Keto form:

Needles from EtOH or Me_2CO. M.p. 223–6° (231°), in Jena glass 245–50°. $FeCl_3 \longrightarrow$ brownish-red col. Conc. $H_2SO_4 \longrightarrow$ yellow col. NaOEt $\longrightarrow$ enol-form.

Dieckmann, *Ber.*, 1916, **49**, 2209.
Abell, *J. Chem. Soc.*, 1912, **101**, 998.
Claisen, *Ann.*, 1896, **291**, 92.

Tribenzylamine

$(C_6H_5{\cdot}CH_2)_3N$

$C_{21}H_{21}N$ MW, 287

Plates or prisms from Et_2O. M.p. 92°. B.p. 230°/13 mm. Sol. hot EtOH, Et_2O. Spar. sol. H_2O. Hot HCl ⟶ dibenzylamine + benzyl chloride. $NaNH_2$ in absence of air or H_2O ⟶ red col.

B,HCl : prisms from EtOH. M.p. 227–8°.

B,HBr : prisms. M.p. 208°.

B,HI : prisms from AcOH. M.p. 185–8° (178°).

B,HNO₃ : cryst. from EtOH. M.p. 120°.

B_2,H_2SO_4 : prisms from EtOH. M.p. 106–7°.

B,Benzenesulphonic acid : needles from H_2O. M.p. 200°.

B,p-Toluenesulphonic acid : m.p. 205·2–207·7°.

Methochloride : needles from H_2O, plates from EtOH. M.p. 202°.

Methiodide : needles from H_2O, plates from EtOH. M.p. 184°.

Ethiodide : needles from H_2O. M.p. 190°.

Picrate : m.p. 191°.

Scheibler, Beiser, Cobler, Schmidt, *Ber.*, 1934, **67**, 1509.
Mason, *J. Chem. Soc.*, 1893, **63**, 1314.

Tribenzyl phosphate.
See under Phosphoric Acid.

Tribromoacetaldehyde.
See Bromal.

Tribromoacetanilide.
See under Tribromoaniline.

Tribromoacetic Acid

$CBr_3{\cdot}COOH$

$C_2HO_2Br_3$ MW, 297

White cryst. M.p. 131°. B.p. 245° decomp. Sol. H_2O, EtOH, Et_2O. Boiling H_2O or EtOH ⟶ bromoform.

Et ester : $C_4H_5O_2Br_3$. MW, 325. B.p. 225°, 148°/73 mm. D_{20}^{20} 2·230. n_D^{13} 1·54377.

Bromide : C_2OBr_4. MW, 360. B.p. 210–15°, 88–90°/12 mm.

Amide : $C_2H_2ONBr_3$. MW, 296. M.p. 121–2°. Sol. Et_2O, hot EtOH. Sublimes.

Nitrile : C_2NBr_3. MW, 278. Oil. B.p. 170°. Sol. EtOH, Et_2O, C_6H_6.

Schäffer, *Ber.*, 1871, **4**, 370.
Müller, U.S.P. 2,057,964, (*Chem. Abstracts*, 1937, **31**, 112).

1 : 1 : 1-Tribromoacetone

$CH_3{\cdot}CO{\cdot}CBr_3$

$C_3H_3OBr_3$ MW, 295

B.p. 255° decomp. NH_3 ⟶ bromoform.

Étard, *Compt. rend.*, 1892, **114**, 754.

ω-Tribromoacetophenone

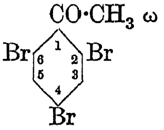
$C_6H_5{\cdot}CO{\cdot}CBr_3$

$C_8H_5OBr_3$ MW, 357

Cryst. from MeOH. M.p. 65–6°. B.p. 174°/14 mm.

Myddleton, Barrett, Seager, *J. Am. Chem. Soc.*, 1930, **52**, 4409.
Aston, Newkirk, Dorsky, Jenkins, *J. Am. Chem. Soc.*, 1942, **64**, 1413.

2 : 4 : 6-Tribromoacetophenone



$C_8H_5OBr_3$ MW, 357

Needles from EtOH.Aq. M.p. 93·5°. Sol. EtOH, Et_2O, C_6H_6.

Fuchs, *Monatsh.*, 1915, **36**, 136.

3 : 4 : 5-Tribromoacetophenone.

Cryst. from EtOH. M.p. 134–5°. Sol. EtOH, C_6H_6. Mod. sol. Et_2O, pet. ether, hot H_2O.

Phenylhydrazone : yellow cryst. from pet. ether. M.p. 129–34° decomp.

Semicarbazone : cryst. from AcOH. M.p. 265° decomp.

Azine : m.p. 300°.

Bruining, *Rec. trav. chim.*, 1922, **41**, 655.

ω : 3 : 5-Tribromoacetophenone.

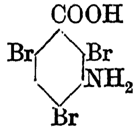
Needles from EtOH. M.p. 85–6°.

Lutz *et al.*, *J. Org. Chem.*, 1947, **12**, 617.

Tribromoacet-toluidide.
See under Tribromotoluidine.

Tribromo-*o*-aminobenzoic Acid.
See Tribromoanthranilic Acid.

2 : 4 : 6-Tribromo-*m*-aminobenzoic Acid



$C_7H_4O_2NBr_3$ MW, 374

Needles from H_2O. M.p. 170·5°. Sol. EtOH. Spar. sol. H_2O. Decomp. on dist. ⟶ 2 : 4 : 6-tribromoaniline.

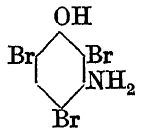
Me ester : $C_8H_6O_2NBr_3$. MW, 388. M.p. 96–7°.

Et ester : $C_9H_8O_2NBr_3$. MW, 402. M.p. 61–2°.

Nitrile : $C_7H_3N_2Br_3$. MW, 355. Needles from EtOH. M.p. 177–8°. Sol. EtOH, $CHCl_3$, Me_2CO.

Sudborough, Karvé, *Chem. Abstracts*, 1920, **14**, 3652.
Sudborough, Lloyd, *J. Chem. Soc.*, 1899, **75**, 589.

2 : 4 : 6-Tribromo-*m*-aminophenol



$C_6H_4ONBr_3$ MW, 346

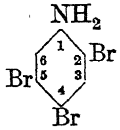
Needles from pet. ether. M.p. 119°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. pet. ether. $FeCl_3$ ⟶ green col.

O : N : N-*Triacetyl* : m.p. 136°.

p-*Toluenesulphonate* : prisms from EtOH. M.p. 146–7°.

Bamberger, *Ber.*, 1915, **48**, 1355.

2 : 4 : 5-Tribromoaniline



$C_6H_4NBr_3$ MW, 330

M.p. 80–1°.

Thomassin, *Compt. rend.*, 1946, **222**, 503.

2 : 4 : 6-Tribromoaniline.

Needles from C_6H_6 or EtOH. M.p. 122° (120°). Sol. Et_2O, hot EtOH. Insol. H_2O. Sn + HCl ⟶ 2 : 4-dibromoaniline. Hot conc. HCl ⟶ 2 : 4 : 6-trichloroaniline.

B,HBr : needles. M.p. 195–6°.

N-*Me* : 2 : 4 : 6-tribromomethylaniline. $C_7H_6NBr_3$. MW, 344. Needles from MeOH. M.p. 39°. B.p. 178–9°/30 mm. N-*Acetyl*: m.p. 101°. N-*Nitroso* : m.p. 91·5–92°.

N-*Di-Me* : 2 : 4 : 6-tribromodimethylaniline. $C_8H_8NBr_3$. MW, 358. Oil. B.p. 301°/750 mm., 134–8°/15 mm. n_D^{20} 1·6244.

N-*Et*: 2:4:6-tribromoethylaniline. $C_8H_8NBr_3$. MW, 358. Needles from AcOH. M.p. 45°.

N-*Formyl* : 2 : 4 : 6-tribromoformanilide. Needles from EtOH. M.p. 221·5°.

N-*Acetyl* : 2 : 4 : 6-tribromoacetanilide. Needles from EtOH. M.p. 232°.

N-*Diacetyl* : needles from EtOH. M.p. 127–8°.

N-*Propionyl* : prisms from EtOH. M.p. 203°.

N-*Benzoyl* : 2 : 4 : 6-tribromobenzanilide. Needles from EtOH. M.p. 204°.

Silberstein, *J. prakt. Chem.*, 1883, **27**, 101.
Asinger, *J. prakt. Chem.*, 1935, **142**, 299.

3 : 4 : 5-Tribromoaniline.

Cryst. from EtOH. M.p. 123°.

N-*Acetyl* : 3 : 4 : 5-tribromoacetanilide. Needles from Et_2O. M.p. 255–6°.

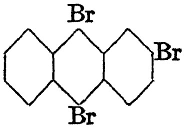
N-*Benzoyl* : 3 : 4 : 5-tribromobenzanilide. Cryst. from EtOH. M.p. 210°.

Asinger, *J. prakt. Chem.*, 1935, **142**, 300.

Tribromoanisole.

See under Tribromophenol.

2 : 9 : 10-Tribromoanthracene

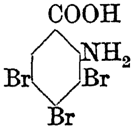


$C_{14}H_7Br_3$ MW, 415

Cryst. from amyl alcohol. M.p. 171°. Very sol. C_6H_6. Spar. sol. EtOH. CrO_3 ⟶ 2-bromoanthraquinone.

Barnett, Cook, *J. Chem. Soc.*, 1925, 1490.
Grandmougin, *Compt. rend.*, 1921, **173**, 1176.

3 : 4 : 5-Tribromoanthranilic Acid (3 : 4 : 5-*Tribromo*-o-*aminobenzoic acid*)

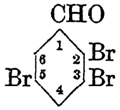


$C_7H_4O_2NBr_3$ MW, 374

Cryst. from EtOH.Aq. Decomp. at 240°.

I.G., D.R.P., 528,115, (*Chem. Zentr.*, 1931, II, 1927).
See also Lesser, Weiss, *Ber.*, 1913, **46**, 3941.

2 : 3 : 5-Tribromobenzaldehyde



$C_7H_3OBr_3$ MW, 343

Cryst. from pet. ether. M.p. 114°. Sol. EtOH. Insol. H_2O.

Blanksma, *Chem. Zentr.*, 1912, II, 1964.

2 : 4 : 6-Tribromobenzaldehyde.

Cryst. from EtOH. M.p. 99°. Sol. EtOH, Et_2O, C_6H_6.

Oxime : needles from EtOH.Aq. M.p. 175°.

Blanksma, *Chem. Zentr.*, 1912, II, 1964.
Lock, Schreckeneder, *Ber.*, 1939, **72**, 511.

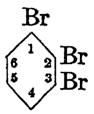
3 : 4 : 5-Tribromobenzaldehyde.

Cryst. from EtOH.Aq. M.p. 109°. Sol. EtOH, C_6H_6. Insol. H_2O.

Oxime : needles from EtOH.Aq. M.p. 172°.
Phenylhydrazone : yellow plates. M.p. 158°.
Semicarbazone : needles from Py. M.p. 314°.
Diacetate : cryst. from EtOH. M.p. 100°.
Azine : yellow needles + $2H_2O$. M.p. 315°.

Janse, *Rec. trav. chim.*, 1921, **40**, 285.
Lock, Schreckeneder, *Ber.*, 1939, **72**, 511.

1 : 2 : 3-Tribromobenzene



$C_6H_3Br_3$ MW, 315

Plates from EtOH. M.p. 87·8°.

Körner, *Gazz. chim. ital.*, 1874, **4**, 408.
Jackson, Gallivan, *Am. Chem. J.*, 1898, **20**, 179.

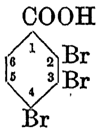
1 : 2 : 4-Tribromobenzene.
Needles with aromatic odour from EtOH. M.p. 44–5°. B.p. 275°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6.

Jackson, Gallivan, *Am. Chem. J.*, 1896, **18**, 241.

1 : 3 : 5-Tribromobenzene.
Needles or prisms from EtOH. M.p. 120°. B.p. 271°. Spar. sol. hot EtOH. Insol. H_2O. NaOMe at 130° ⟶ 3 : 5-dibromophenol.

Coleman, Talbot, *Organic Syntheses*, 1933, XIII, 96.

2 : 3 : 4-Tribromobenzoic Acid



$C_7H_3O_2Br_3$ MW, 359

Needles from C_6H_6. M.p. 197–8°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 511.

2 : 3 : 5-Tribromobenzoic Acid.
Needles from EtOH. M.p. 193–4°. Sol. Et_2O, Me_2CO, hot EtOH. Spar. sol. C_6H_6, pet. ether.
Ba salt: cryst. + $5H_2O$. Very sol. H_2O.
Me ester: $C_8H_5O_2Br_3$. MW, 373. Needles from EtOH.Aq. M.p. 77°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 512.
Blanksma, *Chem. Zentr.*, 1912, II, 1965.

2 : 4 : 5-Tribromobenzoic Acid.
Needles from C_6H_6. M.p. 195–6°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515.

2 : 4 : 6-Tribromobenzoic Acid.
Prisms from H_2O. M.p. 194°.
Me ester: needles from EtOH.Aq M.p. 69°.
Et ester: $C_9H_7O_2Br_3$. MW, 387. M.p. 80°.
Chloride: $C_7H_2OBr_3Cl$. MW, 377·5. Plates from pet. ether. M.p. 48°.
Amide: $C_7H_4ONBr_3$. MW, 358. Prisms. M.p. 195°.
Dimethylamide: $C_9H_8ONBr_3$. MW, 386. Prisms from C_6H_6–pet. ether. M.p. 85–6°.
Nitrile: $C_7H_2NBr_3$. MW, 340. Needles from EtOH. M.p. 128°.
Anilide: needles from EtOH. M.p. 237°.

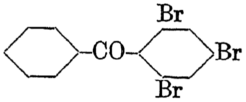
Asinger, *J. prakt. Chem.*, 1935, **142**, 296.
Buning, *Rec. trav. chim.*, 1921, **40**, 327.
Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 516.

3 : 4 : 5-Tribromobenzoic Acid.
Needles from C_6H_6 or EtOH.Aq. M.p. 240°.
Me ester: needles from EtOH. M.p. 154°.
Et ester: needles. M.p. 126°.

Chloride: needles from pet. ether. M.p. 83°.
Amide: needles from EtOH.Aq. M.p. 200°.
Anilide: needles from EtOH. M.p. 220°.

Asinger, *J. prakt. Chem.*, 1935, **142**, 298.
Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 516.

2 : 4 : 6-Tribromobenzophenone

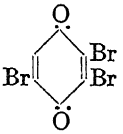


$C_{13}H_7OBr_3$ MW, 419

Cryst. from C_6H_6–pet. ether. M.p. 147°.

Montagne, *Rec. trav. chim.*, 1908, **27**, 353.

Tribromo-*p*-benzoquinone (*Tribromoquinone*)

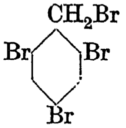


$C_6HO_2Br_3$ MW, 345

Yellow plates from EtOH. M.p. 147°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Sublimes. NaOH ⟶ green col.

Sarauw, *Ann.*, 1881, **209**, 120.

2:4:6-Tribromobenzyl bromide (α:2:4:6-*Tetrabromotoluene*)



$C_7H_4Br_4$ MW, 408

Needles from EtOH or AcOH. M.p. 75°. B.p. 202°/18 mm.

Asinger, *J. prakt. Chem.*, 1935, **142**, 296.

Tribromobenzyl cyanide.
See under Tribromophenylacetic Acid.

1 : 1 : 2-Tribromobutane

$CH_3{\cdot}CH_2{\cdot}CHBr{\cdot}CHBr_2$

$C_4H_7Br_3$ MW, 295

Yellow oil with camphor-like odour. B.p. 216·2°, 98°/14 mm. D_4^{15} 2·1913. Darkens on heating at atm. press.

Kaufmann, Schweitzer, *Ber.*, 1922, **55**, 264.

1 : 2 : 2-Tribromobutane

$CH_3{\cdot}CH_2{\cdot}CBr_2{\cdot}CH_2Br$

$C_4H_7Br_3$ MW, 295

B.p. 213·8°, 112–15°/40 mm., 90·1°/14 mm. D_4^{15} 2·1761.

Lépingle, *Bull. soc. chim.*, 1926, **39**, 741.

1 : 2 : 3-Tribromobutane

$CH_3{\cdot}CHBr{\cdot}CHBr{\cdot}CH_2Br$

$C_4H_7Br_3$ MW, 295

B.p. 110–13°/19 mm., 100–1°/14 mm., 94–6°/6 mm. D_4^{16} 2·190. n_D^{15} 1·5691.

Slobodin, *Chem. Abstracts*, 1935, **29**, 4732.
Delaby, *Compt. rend.*, 1923, **176**, 589.

1 : 2 : 4-Tribromobutane

$BrCH_2·CH_2·CHBr·CH_2Br$

$C_4H_7Br_3$ MW, 295

B.p. 115–17°/10 mm. D_0^{18} 2·234. n_D^{18} 1·574.

Pariselle, *Ann. chim. phys.*, 1911, **24**, 323.

2 : 2 : 3-Tribromobutane

$CH_3·CHBr·CBr_2·CH_3$

$C_4H_7Br_3$ MW, 295

B.p. 206·5°, 83–4°/11·5 mm. D_4^{15} 2·1806.

Lépingle, *Bull. soc. chim.*, 1926, **39**, 741.

1 : 3 : 4-Tribromo-*sec.*-*n*-butyl Alcohol (*Erythritol tribromohydrin*, 1 : 3 : 4-*tribromo*-2-*hydroxybutane*)

$BrCH_2·CHBr·CH(OH)·CH_2Br$

$C_4H_7OBr_3$ MW, 311

B.p. 148–50°/14 mm. D_4^{20} 2·3097. n_D^{20} 1·5858.
Acetate: b.p. 146°/10 mm. D_4^{20} 2·0349. n_D^{20} 1·5418.

Pariselle, *Ann. chim. phys.*, 1911, **24**, 405.
Petrov, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 142.

1 : 1 : 1 - Tribromo - *tert.* - butyl Alcohol (*Brometone*)

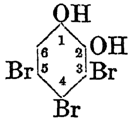
$(CH_3)_2C(OH)·CBr_3$

$C_4H_7OBr_3$ MW, 311

Cryst. with camphor-like odour from EtOH.Aq. M.p. 167–76°.
Acetyl: cryst. from EtOH. M.p. 43–4°.
Propionyl: cryst. from EtOH. M.p. 27°.
Butyryl: b.p. 144–5°/13 mm.

Aldrich, *J. Am. Chem. Soc.*, 1911, **33**, 387.

3 : 4 : 5-Tribromocatechol



$C_6H_3O_2Br_3$ MW, 347

Cryst. + $1H_2O$. M.p. 144°. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. $CHCl_3$, pet. ether. Insol. H_2O.
Di-Me ether: 3 : 4 : 5 - tribromoveratrol. $C_8H_7O_2Br_3$. MW, 375. Needles. M.p. 86–7°. Sol. $CHCl_3$, C_6H_6. Spar. sol. cold EtOH.
Diacetyl: cryst. M.p. 120°.

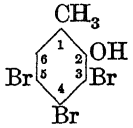
Frejka, Šefránek, *Chem. Zentr.*, 1936, I, 2338.
Chem. Fabrik, von Heyden, D.R.P., 207,544, (*Chem. Zentr.*, 1909, I, 1283); 215,337, (*Chem. Zentr.*, 1909, II, 1710).

3 : 4 : 6-Tribromocatechol.

Needles. M.p. 106°.
Di-Me ether: needles. M.p. 69°.
Diacetyl: needles. M.p. 141°.

Kohn, Steiner, *J. Org. Chem.*, 1947, **12**, 30.

3 : 4 : 5-Tribromo-*o*-cresol



$C_7H_5OBr_3$ MW, 345

Needles from petrol. M.p. 89°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Spar. sol. petrol.
Acetyl: plates from AcOH. M.p. 106–7°.

Kohn, Aron, *Monatsh.*, 1929, **53**, 49.
Janney, *Ann.*, 1913, **398**, 367.

3 : 5 : 6-Tribromo-*o*-cresol.

Needles from petrol. M.p. 91°. Sol. Et_2O, AcOH, C_6H_6. Spar. sol. petrol. $NaNO_2$ in AcOH ⟶ 3 : 4-dibromo-6-nitro-*o*-cresol.
Me ether: $C_8H_7OBr_3$. MW, 359. Needles from EtOH. M.p. 71°. B.p. 308–11°/745 mm.
Acetyl: cryst. from AcOH. M.p. 76–7°.
Benzoyl: prisms from C_6H_6. M.p. 133°.

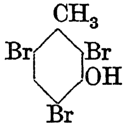
Kohn, Aron, *Monatsh.*, 1929, **53**, 54.

4 : 5 : 6-Tribromo-*o*-cresol.

Needles from pet. ether. M.p. 106°.
Me ether: cryst. from EtOH. M.p. 105°. B.p. 320°.

Kohn, Soltész, *Monatsh.*, 1925, **46**, 250.

2 : 4 : 6-Tribromo-*m*-cresol

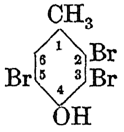


$C_7H_5OBr_3$ MW, 345

Needles from EtOH or pet. ether. M.p. 81·5–82°. Cryst. + 1AcOH from AcOH. Ox. ⟶ 3 : 5-dibromotoluquinone.
Et ether: $C_9H_9OBr_3$. MW, 373. Needles from EtOH. M.p. 36°.
Acetyl: needles from EtOH. M.p. 68°.
Benzoyl: needles from EtOH. M.p. 84–5°.
Benzenesulphonyl: plates from EtOH. M.p. 117–117·5°.
p-*Toluenesulphonyl*: plates from EtOH. M.p. 113–14°.

Huston, Peterson, *J. Am. Chem. Soc.*, 1933, **55**, 3882.
Jost, Richter, *Ber.*, 1923, **56**, 122.
van Erp, *Rec. trav. chim.*, 1911, **30**, 302.
Claus, Hirsch, *J. prakt. Chem.*, 1889, **39**, 59.

2 : 3 : 5-Tribromo-*p*-cresol



$C_7H_5OBr_3$ MW, 345

Needles from petrol. M.p. 102°. Sol. EtOH, Et_2O, AcOH, C_6H_6, hot Na_2CO_3.Aq. Spar. sol. petrol.

Acetyl: needles from ligroin. M.p. 77°.

Zincke, Wiederhold, *Ann.*, 1902, **320**, 205.

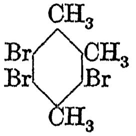
2 : 3 : 6-Tribromo-*p*-cresol.

Benzoyl: plates from Me_2CO. M.p. 120°.

p-*Nitrobenzoyl*: plates from Me_2CO–$CHCl_3$. Softens at 156°, m.p. 159–60°.

Jadhav, Rangwala, *Chem. Zentr.*, 1935, I, 2976.

3 : 5 : 6-Tribromo-ψ-cumene (3 : 5 : 6-*Tribromo*-1 : 2 : 4-*trimethylbenzene*)

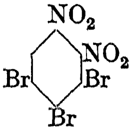


$C_9H_9Br_3$ MW, 357

Cryst. M.p. 229–30°. Sol. boiling toluene, hot AcOH. Spar. sol. hot EtOH.

Fittig, Laubinger, *Ann.*, 1869, **151**, 267.

3 : 4 : 5-Tribromo-*o*-dinitrobenzene

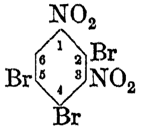


$C_6HO_4N_2Br_3$ MW, 405

Plates or prisms. M.p. 162–4°. Sol. AcOH, $CHCl_3$, C_6H_6, EtOH, Et_2O, hot ligroin. Insol. H_2O. Boiling dil. NaOH ⟶ 4 : 5 : 6-tribromo-*o*-nitrophenol.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 586.

2 : 4 : 5-Tribromo-*m*-dinitrobenzene



$C_6HO_4N_2Br_3$ MW, 405

Yellow scales. M.p. 135·5°. Sol. hot EtOH, Et_2O, CS_2. Spar. sol. cold EtOH. Red. ⟶ 5-bromo-*m*-phenylenediamine.

Jackson, Gallivan, *Ber.*, 1895, **28**, 190.

2 : 4 : 6-Tribromo-*m*-dinitrobenzene.

Colourless prisms or needles from EtOH. M.p. 192°. Very sol. Et_2O, C_6H_6, CS_2. Sol. hot EtOH. Spar. sol. cold EtOH. Insol. H_2O. Red. ⟶ 2 : 4 : 6-tribromo-*m*-phenylenediamine. Zn + HCl ⟶ *m*-phenylenediamine.

Jackson, Koch, *Am. Chem. J.*, 1899, **21**, 519.

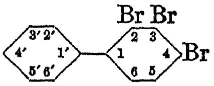
4 : 5 : 6-Tribromo-*m*-dinitrobenzene.

Cryst. from EtOH. M.p. 150–1°. Sol. Me_2CO, AcOH, $CHCl_3$, C_6H_6. Spar. sol. EtOH, ligroin. Alc. NH_3 at 100° ⟶ 5 : 6-dibromo-2 : 4-dinitroaniline.

van de Bunt, *Rec. trav. chim.*, 1929, **48**, 128.

Jackson, Earle, *Am. Chem. J.*, 1901, **26**, 51.

2 : 3 : 4-Tribromodiphenyl



$C_{12}H_7Br_3$ MW, 391

Needles. M.p. 225–7°.

Bellavita, *Chem. Zentr.*, 1936, I, 2342.

2 : 4 : 5-Tribromodiphenyl.

Prisms. M.p. 68°.

Bellavita, *Chem. Zentr.*, 1936, I, 2341.

2 : 4 : 6-Tribromodiphenyl.

Needles from MeOH. M.p. 64°. B.p. 190–8°/4 mm.

Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3008.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

2 : 5 : 2′-Tribromodiphenyl.

M.p. 77–8°.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

2 : 5 : 3′-Tribromodiphenyl.

B.p. 213–16°/6 mm.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

2 : 5 : 4′-Tribromodiphenyl.

Yellow needles from EtOH. M.p. 76°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 643.

2 : 3′ : 4′-Tribromodiphenyl.

Needles from EtOH. M.p. 91°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 644.

2 : 3′ : 5′-Tribromodiphenyl.

M.p. 68–9°.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

3 : 4 : 4′-Tribromodiphenyl.

Prisms. M.p. 102°.

Bellavita, *Chem. Zentr.*, 1936, I, 2342.

3 : 5 : 3′-Tribromodiphenyl.

M.p. 112–13°.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

3 : 5 : 4′-Tribromodiphenyl.

M.p. 102–3°.

Case, *J. Am. Chem. Soc.*, 1939, **61**, 3487.

α : β : 4-Tribromodiphenylethane.

See under p-Bromostilbene.

1 : 1 : 2-Tribromoethane

$CH_2Br{\cdot}CHBr_2$

$C_2H_3Br_3$ MW, 267

M.p. – 26°. B.p. 187–8°/721 mm., 83°/18 mm. D_4^{20} 2·57896. n_D^{20} 1·58902. Alc. KOH ⟶ 1 : 1-dibromoethylene + bromoacetylene + acetylene.

Wurtz, *Ann.*, 1857, **104**, 243.

2 : 2 : 2-Tribromoethyl Alcohol (*Tribromoethanol, Avertin*)

$CBr_3{\cdot}CH_2OH$

$C_2H_3OBr_3$ MW, 283

Needles or prisms. M.p. 80°. B.p. 92–3°/10 mm. Sol. EtOH, Et_2O, C_6H_6, warm pet. ether. Spar. sol. H_2O. Used in anæsthesia.

Benzoyl: m.p. 35·5–36·5°.
o-*Nitrobenzoyl*: m.p. 117–18°.
m-*Nitrobenzoyl*: m.p. 82·5–83°.
p-*Nitrobenzoyl*: m.p. 100–1°.
Chloroformyl: b.p. 103°/10 mm.
Carbonate: m.p. 112–13°.
Urethane: prisms from EtOH. M.p. 86–7°. Very sol. H_2O. Sol. EtOH, Et_2O.
Phenylurethane: m.p. 107–8°.
p-*Bromophenylurethane*: m.p. 116–17°.

I.G., D.R.P., 565,157, (*Chem. Zentr.*, 1933, I, 1514); B.P. 370,490, (*Chem. Zentr.*, 1932, II, 3303).
Willstätter, Duisberg, *Ber.*, 1923, **56**, 2284.
Carli, Mangini, *Chem. Abstracts*, 1937, **31**, 7396.
Edwards, *Chem. Abstracts*, 1946, **40**, 3232.

Tribromoethylene

$CHBr{:}CBr_2$

C_2HBr_3 MW, 265

B.p. 162·5°, 75°/15 mm. $D^{20·5}$ 2·708. n_D^{25} 1·62475. O ⟶ dibromoacetyl bromide. In air ⟶ pentabromoethane. Zn + EtOH ⟶ acetylene + bromoacetylene.

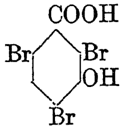
Hg comp.: prisms from $CHCl_3$. M.p. 141°.

Dehn, *J. Am. Chem. Soc.*, 1912, **34**, 286.

Tribromoethylidene diacetate.
See under Bromal.

Tribromohydrin.
See 1 : 2 : 3-Tribromopropane.

2 : 4 : 6-Tribromo-*m*-hydroxybenzoic Acid



$C_7H_3O_3Br_3$ MW, 375

Needles + ½H_2O from H_2O. Anhyd. at 100°. M.p. 146–7°. Sol. EtOH, Et_2O, AcOH. Mod. sol. $CHCl_3$. Insol. C_6H_6. Boiling HI ⟶ *m*-hydroxybenzoic acid.

Me ether: 2 : 4 : 6-tribromo-*m*-methoxybenzoic acid. $C_8H_5O_3Br_3$. MW, 389. Cryst. from AcOH.Aq. M.p. 154°.
Me ester: $C_8H_5O_3Br_3$. MW, 389. Plates and needles from ligroin. M.p. 119–21°. Sol. most org. solvents.
Amide: $C_7H_4O_2NBr_3$. MW, 374. M.p. 221°.
Nitrile: $C_7H_2ONBr_3$. MW, 356. Yellow needles from ligroin. M.p. 168°. Spar. sol. ligroin. *Acetyl*: yellowish-white plates from EtOH.Aq. M.p. 156–8°.
Acetyl: cryst. from MeOH or $CHCl_3$–pet. ether. M.p. 192–3°.

Bull, Ross, Fuson, *J. Am. Chem. Soc.*, 1935, **57**, 764.
Luck, Hosaeus, *Monatsh.*, 1933, **62**, 186.
Leulier, Pinet, *Bull. soc. chim.*, 1927, **41**, 1362.
Werner, *Bull. soc. chim.*, 1886, **46**, 276.

Tribromohydroxybenzyl bromide.
See Tetrabromocresol.

1 : 1 : 2-Tribromoisobutane

$(CH_3)_2CBr{\cdot}CHBr_2$

$C_4H_7Br_3$ MW, 295

B.p. 205–6° decomp., 121–4°/57 mm., 110–14°/15 mm. D_4^{21} 2·0169.

Norton, Williams, *Am. Chem. J.*, 1887, **9**, 89.

1 : 2 : 3-Tribromoisobutane

$BrH_2C \choose CH_3$ $>CBr{\cdot}CH_2Br$

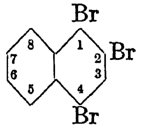
$C_4H_7Br_3$ MW, 295

B.p. 173–83°/235 mm., 108–9°/18 mm. D_4^{14} 2·2106. n_D^{14} 1·57012.

Mereshkowski, *Chem. Zentr.*, 1914, I, 2160.
Norton, Williams, *Am. Chem. J.*, 1887, **9**, 88.

Tribromomethane.
See Bromoform.

1 : 2 : 4-Tribromonaphthalene



$C_{10}H_5Br_3$ MW, 365

Needles. M.p. 113–14°. Very sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Mod. sol. EtOH, Me_2CO. Ox. ⟶ phthalic acid.

Prager, *Ber.*, 1885, **18**, 2164.

1 : 2 : 6-Tribromonaphthalene.

Needles from EtOH. M.p. 118°. Very sol. EtOH, Et_2O, ligroin. Slowly volatile in steam. Sublimes.

Claus, Philipson, *J. prakt. Chem.*, 1891, **43**, 53.

1 : 3 : 6-Tribromonaphthalene.
Needles from EtOH, plates from H_2O. M.p. 98° (86·5°). Sol. most. org. solvents. Sublimes.

Franzen, Stäuble, *J. prakt. Chem.*, 1920, **101**, 58.
Claus, Jäck, *J. prakt. Chem.*, 1898, **57**, 17.

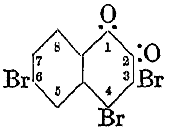
1 : 4 : 5-Tribromonaphthalene.
Needles. M.p. 86°. Very sol. EtOH.

Salkind, Belikoff, *Ber.*, 1931, **64**, 958.
John, *Bull. soc. chim.*, 1877, **28**, 515.

1 : 4 : 6-Tribromonaphthalene.
Needles from EtOH. M.p. 86–7°. Very sol. EtOH, Et_2O.

Salkind, Belikoff, *Ber.*, 1931, **64**, 959.
Glaser, *Ann.*, 1865, **135**, 43.

3 : 4 : 6-Tribromo-1 : 2-naphthoquinone



$C_{10}H_3O_2Br_3$ MW, 395

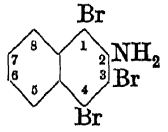
Red needles from C_6H_6. M.p. 193°.
Quinoxaline with o-*phenylenediamine* : yellow needles from AcOH. M.p. 250°.

Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 274.

3 : 5 : 6-Tribromo-1 : 2-naphthoquinone.
Red needles from C_6H_6–petrol. M.p. 184°. Mod. sol. EtOH, AcOH, pet. ether.
Quinoxaline with o-*phenylenediamine* : yellow needles from toluene. M.p. 271°.

Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 282.

1 : 3 : 4-Tribromo-2-naphthylamine



$C_{10}H_6NBr_3$ MW, 380

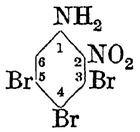
Pale straw coloured cryst. M.p. 163°.

Hodgson, Hathway, *J. Chem. Soc.*, 1944, 21.

1 : 3 : 6-Tribromo-2-naphthylamine.
Pale red cryst. from EtOH. M.p. 143°. Very sol. Et_2O, $CHCl_3$. Spar. sol. EtOH, pet. ether. Ox. ⟶ 4-bromophthalic acid.
N-*Acetyl* : 1 : 3 : 6-tribromo-2-acetnaphthalide. Needles from EtOH. M.p. 250–1°.
N-*Diacetyl* : plates from EtOH. M.p. 159°.
N-p-*Toluenesulphonyl* : needles from AcOH. M.p. 184°.

Bell, *J. Chem. Soc.*, 1932, 2734.
Franzen, Stäuble, *J. prakt. Chem.*, 1920, **101**, 61.

3 : 4 : 5-Tribromo-*o*-nitroaniline



$C_6H_3O_2N_2Br_3$ MW, 375

Yellow needles. M.p. 134°. Sol. EtOH. Spar. sol. hot H_2O.
N-*Acetyl* : 3 : 4 : 5-tribromo-2-nitroacetanilide. Needles from EtOH or C_6H_6, prisms from AcOEt. M.p. 229–30°.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 581.

4 : 5 : 6-Tribromo-*o*-nitroaniline.
Yellow needles. M.p. 166°. Very sol. Me_2CO, $CHCl_3$, C_6H_6. Sol. MeOH, Et_2O, AcOH. Spar. sol. EtOH. Insol. H_2O, ligroin.
N-*Me* : $C_7H_5O_2N_2Br_3$. MW, 389. M.p. 128°.
N-*Et* : $C_8H_7O_2N_2Br_3$. MW, 403. M.p. 130°.
N-*Phenyl* : 4 : 5 : 6-tribromo-2-nitrodiphenylamine. $C_{12}H_7O_2N_2Br_3$. MW, 451. Light red prisms or yellow needles. M.p. 138–9°. Sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. EtOH, AcOH. Insol. H_2O, ligroin.
N-*Acetyl* : needles. M.p. 221°.

Jackson, Fiske, *Am. Chem. J.*, 1903, **30**, 74.
Blanksma, *Rec. trav. chim.*, 1902, 21, 414.

2 : 4 : 6-Tribromo-*m*-nitroaniline.
Yellow needles from EtOH. M.p. 120·5°. Very sol. EtOH.
N-*Acetyl* : 2 : 4 : 6-tribromo-3-nitroacetanilide. Cryst. from EtOH or AcOH, needles from C_6H_6. M.p. 208–9° (216–17°).
N-*Diacetyl* : cryst. from EtOH. M.p. 168–9° (175–6°).
N-*Chloro* : $C_8H_4O_3N_2ClBr_3$. MW, 409·5. Prisms from $CHCl_3$–pet. ether. M.p. 159°.

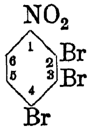
Jackson, Jones, *Am. Chem. J.*, 1913, **49**, 48.

2 : 3 : 6-Tribromo-*p*-nitroaniline.
Yellow needles. M.p. 159°. Spar. sol. cold EtOH. Insol. H_2O. Slowly volatile in steam.

Orton, *J. Chem. Soc.*, 1902, **81**, 499.

Tribromonitroanisole.
See under Tribromonitrophenol.

2 : 3 : 4-Tribromonitrobenzene



$C_6H_2O_2NBr_3$ MW, 360

Cryst. from EtOH. M.p. 85·4°. Very sol. Et_2O, AcOEt, C_6H_6. Mod. sol. EtOH. Slowly volatile in steam. Sublimes with difficulty.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 583.

2 : 3 : 5-Tribromonitrobenzene.
Needles. M.p. 81° (119·5°). Volatile in steam. Red. ⟶ 2 : 3 : 5-tribromoaniline.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 58.

2 : 3 : 6-Tribromonitrobenzene.
Colourless plates or prisms from EtOH–Et_2O. Sublimes at 185° without melting. Very spar. sol. EtOH.

Körner, *Jahresber. Fortschr. Chem.*, 1875, 314.

2 : 4 : 5-Tribromonitrobenzene.
Needles from EtOH. M.p. 93·5°. Sol. Et_2O, hot EtOH, CS_2. Dist. without decomp. Volatile in steam. Sublimes. Red. ⟶ 2 : 4 : 5-tribromoaniline.

Körner, *Jahresber. Fortschr. Chem.*, 1875, 313.

2 : 4 : 6-Tribromonitrobenzene.
Colourless prisms from $CHCl_3$. M.p. 124·5°. B.p. about 177°/11 mm. Sol. Et_2O, boiling AcOH, $CHCl_3$. Spar. sol. boiling EtOH. Red. ⟶ 2 : 4 : 6-tribromoaniline.

Orton, *J. Chem. Soc.*, 1903, **83**, 806.
Hantzsch, Blagden, *Ber.*, 1900, **33**, 2553.

3 : 4 : 5-Tribromonitrobenzene.
Colourless cryst. from EtOH–Et_2O. M.p. 112°. Volatile in steam. Sublimes. Red. ⟶ 3 : 4 : 5-tribromoaniline. NaOH ⟶ 2 : 6-dibromo-*p*-nitrophenol.

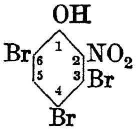
Asinger, *J. prakt. Chem.*, 1935, **142**, 299.
Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 585.

Tribromonitrodiphenylamine.
See under Tribromonitroaniline.

Tribromonitromethane.
See Bromopicrin.

Tribromonitrophenetole.
See under Tribromonitrophenol.

3 : 4 : 6-Tribromo-*o*-nitrophenol



$C_6H_2O_3NBr_3$ MW, 376

Pale yellow plates from H·COOH.Aq. M.p. 127°.
Me ether: 3 : 4 : 6-tribromo-*o*-nitroanisole. $C_7H_4O_3NBr_3$. MW, 390. Colourless prisms from H·COOH.Aq. M.p. 72°.

Hodgson, Walker, Smith, *J. Chem. Soc.*, 1933, 1055.

4 : 5 : 6-Tribromo-*o*-nitrophenol.
Yellow prisms from C_6H_6 or MeOH. M.p. 123°. Very sol. Me_2CO, hot C_6H_6, $CHCl_3$. Sol. EtOH, Et_2O, AcOH. Insol. cold C_6H_6.
Me ether: 4 : 5 : 6-tribromo-*o*-nitroanisole. Needles from EtOH or H·COOH. M.p. 109°. Very sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. EtOH, AcOH, ligroin. Insol. H_2O.
Et ether: 4 : 5 : 6-tribromo-*o*-nitrophenetole. $C_8H_6O_3NBr_3$. MW, 404. Cryst. M.p. 74°. Rapidly turns brown in air.

Hodgson, Walker, Smith, *J. Chem. Soc.*, 1933, 1055.
Jackson, Fiske, *Am. Chem. J.*, 1903, **30**, 71.

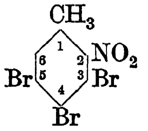
2 : 4 : 6-Tribromo-*m*-nitrophenol.
Cryst. from ligroin. M.p. 89–90°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. warm H_2O. Decomposes cold aq. carbonates. Forms spar. sol. Ag and Ba salts. Red. ⟶ *m*-aminophenol.
Me ether: 2 : 4 : 6-tribromo-*m*-nitroanisole. $C_7H_4O_3NBr_3$. MW, 390. Plates from EtOH. M.p. 82°.
Et ether: 2 : 4 : 6-tribromo-*m*-nitrophenetole. $C_8H_6O_3NBr_3$. MW, 404. Prisms from Et_2O. M.p. 79°. Very sol. Et_2O.
Propionyl: scales from EtOH.Aq. M.p. 70–1°.
p-*Toluenesulphonyl*: needles from EtOH. M.p. 146–7°.

Henley, Turner, *J. Chem. Soc.*, 1930, 935.
Bamberger, *Ber.*, 1915, **48**, 1344.

2 : 3 : 6-Tribromo-*p*-nitrophenol.
Pale yellow needles from hot H_2O. M.p. 151° decomp.

Hodgson, Smith, *J. Chem. Soc.*, 1932, 505.

3 : 4 : 5-Tribromo-*o*-nitrotoluene



$C_7H_4O_2NBr_3$ MW, 374

Cryst. from EtOH. M.p. 104–5°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515.

3 : 5 : 6-Tribromo-*o*-nitrotoluene.
Cryst. from EtOH. M.p. 88·5–90° (93°). Sol. hot EtOH, C_6H_6. Spar. sol. pet. ether.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 512.

2 : 4 : 6-Tribromo-*m*-nitrotoluene.
Cryst. M.p. 74–75·5°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515.

2 : 5 : 6-Tribromo-*m*-nitrotoluene.
Yellow needles from EtOH. M.p. 91–2°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 514.

2 : 3 : 5-Tribromo-*p*-nitrotoluene.
Cryst. from EtOH. M.p. 67–8°.

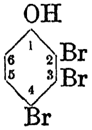
Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 512

2 : 3 : 6-Tribromo-*p*-nitrotoluene.
Cryst. from AcOH. M.p. 106·5–107°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 514.

Tribromophenetole.
See under Tribromophenol.

2 : 3 : 4-Tribromophenol



$C_6H_3OBr_3$ MW, 331

Prisms from H·COOH.Aq. M.p. 95°.

Me ether: 2 : 3 : 4-tribromoanisole. $C_7H_5OBr_3$. MW, 345. M.p. 106°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.

2 : 3 : 5-Tribromophenol.
Needles or plates from H_2O or ligroin. M.p. 94–5°. Very sol. EtOH, Et_2O, Me_2CO, hot ligroin, alkalis. Spar. sol. H_2O. Volatile in steam. $FeCl_3$ ⟶ brownish-violet col.

Me ether: 2 : 3 : 5-tribromoanisole. M.p. 82°. B.p. 305–12°.

Bamberger, Kraus, *Ber.*, 1906, **39**, 4251.
Kohn, Karlin, *Monatsh.*, 1927, **48**, 599.

2 : 4 : 5-Tribromophenol.
Needles from dichloroethane–pet. ether. M.p. 87°.

Me ether: 2 : 4 : 5-tribromoanisole. Needles. M.p. 105°. B.p. 306–9°/775 mm.

Benzoyl: m.p. 99°.

o-*Bromo*-p-*toluenesulphonyl*: plates from EtOH. M.p. 107–8°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.
Turner, Henley, *J. Chem. Soc.*, 1930, 933.

2 : 4 : 6-Tribromophenol (*Bromol*).
Needles from EtOH, prisms from C_6H_6, cryst. + 1AcOH from AcOH. M.p. 94°. Sol. 14,000 parts H_2O at 15°. Very sol. EtOH. Sublimes. $NaNO_2$ in AcOH ⟶ 4 : 6-dibromo-*o*-nitrophenol. Forms spar. sol. NH_4 salt. Stronger antiseptic than phenol or thymol.

Me ether: 2 : 4 : 6-tribromoanisole. $C_7H_5OBr_3$. MW, 345. Needles from EtOH. M.p. 88°. Sol. 100 parts H_2O at 15°.

Et ether: 2 : 4 : 6-tribromophenetole. $C_8H_7OBr_3$. MW, 359. Prisms from EtOH. M.p. 72–3°.

Propyl ether: $C_9H_9OBr_3$. MW, 373. Needles from EtOH. M.p. 33–4°.

Allyl ether: $C_9H_7OBr_3$. MW, 371. Needles. M.p. 77°.

p-*Nitrobenzyl ether*: $C_{13}H_8O_3NBr_3$. MW, 466. M.p. 163·5°.

Acetyl: colourless plates or needles from EtOH. M.p. 87°.

Propionyl: needles. M.p. 65°.

Benzoyl: m.p. 81°.

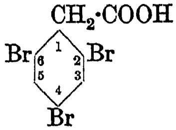
p-*Bromobenzenesulphonyl*: cryst. from EtOH. M.p. 139–40°.

p-*Toluenesulphonyl*: cryst. from EtOH. M.p. 113°.

p-*Iodophenylurethane*: m.p. 190–1°.

van Erp, *Rec. trav. chim.*, 1911, **30**, 280.
Orton, Coates, Burdett, *J. Chem. Soc.*, 1907, **91**, 47.
Körner, *Ann.*, 1866, **137**, 203.
Sah, Young, *Rec. trav. chim.*, 1940, **59**, 357.
Raiford, Le Rosen, *J. Am. Chem. Soc.*, 1946, **68**, 397.

2 : 4 : 6-Tribromophenylacetic Acid



$C_8H_5O_2Br_3$ MW, 373

M.p. 157–8°.

Amide: $C_8H_6ONBr_3$. MW, 372. M.p. 162–3°.

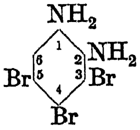
Nitrile: 2 : 4 : 6-tribromobenzyl cyanide. $C_8H_4NBr_3$. MW, 354. M.p. 138–9°. Sol. EtOH. Insol. H_2O.

Henraut, *Chem. Zentr.*, 1924, II, 1342.

3 : 4 : 5-Tribromophenylacetic Acid.
Me ester: m.p. 78–9°.

Corse *et al.*, *J. Am. Chem. Soc.*, 1948, **70**, 2837.

3 : 4 : 5-Tribromo-*o*-phenylenediamine

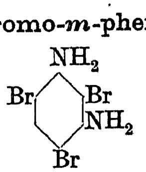


$C_6H_5N_2Br_3$ MW, 345

Cryst. from EtOH. M.p. 91°. Very sol. Et_2O, Me_2CO. Sol. EtOH, AcOH, C_6H_6. Insol. H_2O, ligroin. Turns brown in air.

Jackson, *Am. Chem. J.* 1903, **30**, 78.

2 : 4 : 6-Tribromo-*m*-phenylenediamine



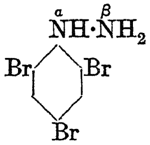
$C_6H_5N_2Br_3$ MW, 345

Needles from EtOH. M.p. 158°. Very sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Zn + HCl ⟶ *m*-phenylenediamine.

1 : 3-N-*Diacetyl*: plates from AcOH. Does not melt below 330°.

Jackson, Calvert, *Am. Chem. J.*, 1896, **18**, 470.

2 : 4 : 6-Tribromophenylhydrazine



$C_6H_5N_2Br_3$ MW, 345

Needles from pet. ether. M.p. 146°. Sol. boiling AcOH. Spar. sol. boiling EtOH.

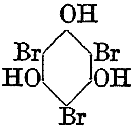
β-N-*Acetyl*: prisms from MeOH. M.p. 188°.

α : β-N-*Diacetyl*: needles from EtOH. M.p. 144–5°.

β-N-*Benzoyl*: yellow needles from EtOH. M.p. 172°.

Chattaway, Vonderwahl, *J. Chem. Soc.*, 1915, **107**, 1507.

Tribromophloroglucinol



$C_6H_3O_3Br_3$ MW, 363

Cryst. + $3H_2O$ from H_2O. M.p. anhyd. 152–3° (149–50°). Sn + HCl ⟶ phloroglucinol.

Me ether: $C_7H_5O_3Br_3$. MW, 377. Needles from C_6H_6. M.p. 123°. Very sol. H_2O, EtOH, $CHCl_3$. *Diacetyl*: needles from EtOH. M.p. 112–14°.

Tri-Me ether: $C_9H_9O_3Br_3$. MW, 405. Needles from EtOH. M.p. 145°.

Di-Et ether: $C_{10}H_{11}O_3Br_3$. MW, 419. Needles from AcOH.Aq. M.p. 63–5°.

Tri-Et ether: $C_{12}H_{15}O_3Br_3$. MW, 447. Needles from AcOH. M.p. 102–4°.

Monoacetyl: needles from C_6H_6–pet. ether. M.p. 169°.

Triacetyl: needles from EtOH. M.p. 181–3°.

Herzig, Kaserer, *Monatsh.*, 1902, **23**, 577.
Perkin, Simonsen, *J. Chem. Soc.*, 1905, **87**, 863.

1 : 1 : 2-Tribromopropane

$CH_3·CHBr·CHBr_2$

$C_3H_5Br_3$ MW, 281

B.p. 200–1°, 100–3°/20 mm., 83°/6 mm. D_4^{20} 2·356. n_D^{20} 1·573983.

Mereshkowsky, *Ann.*, 1923, **431**, 239.
Bachman, *J. Am. Chem. Soc.*, 1935, **57**, 1090.

1 : 2 : 2-Tribromopropane

$CH_3·CBr_2·CH_2Br$

$C_3H_5Br_3$ MW, 281

B.p. 190–1°, 80·6°/20 mm. D^{12} 2·23. n_D^{20} 1·566963.

Mereshkowsky, *Ann.*, 1923, **431**, 241.

1 : 2 : 3-Tribromopropane (*Glycerol tribromohydrin, tribromohydrin*)

$BrCH_2·CHBr·CH_2Br$

$C_3H_5Br_3$ MW, 281

M.p. 16–17°. B.p. 219–21°, 115–20°/30 mm., 100–3°/18 mm. $D_{15}^{18·5}$ 2·3955. n_D^{18} 1·584. Zn dust + EtOH ⟶ propylene + allyl bromide. Alc. KOH ⟶ ethyl propargyl ether. Ag acetate ⟶ triacetin.

Johnson, McEwen, *Organic Syntheses*, Collective Vol. I, 507.

1 : 1 : 2-Tribromopropionic Acid

$CH_2Br·CBr_2·COOH$

$C_3H_3O_2Br_3$ MW, 311

Cryst. from CS_2. M.p. 95° (92°). Very sol. EtOH, Et_2O, CS_2, C_6H_6. Spar. sol. cold H_2O. Baryta ⟶ 1 : 2-dibromoacrylic acid.

Et ester: $C_5H_7O_2Br_3$. MW, 339. Liq. with aromatic odour. B.p. 140–2°/30 mm. D^{23} 2·084. n_D^{23} 1·532.

Michael, Norton, *Am. Chem. J.*, 1880, **2**, 18.
Berlande, *Bull. soc. chim.*, 1925, **37**, 1390.

1 : 2 : 2-Tribromopropionic Acid

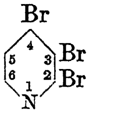
$CHBr_2·CHBr·COOH$

$C_3H_3O_2Br_3$ MW, 311

Leaflets. M.p. 118°. Very sol. EtOH, Et_2O. Sol. $CHCl_3$, CS_2, C_6H_6, ligroin, hot H_2O. Baryta ⟶ 1 : 2-dibromoacrylic acid.

Hill, Andrews, *Am. Chem. J.*, 1882, **4**, 180.

2 : 3 : 4-Tribromopyridine



$C_5H_2NBr_3$ MW, 316

M.p. 84–5°.

Wibaut, Hertog, *Rec. trav. chim.*, 1945, **64**, 55.

2 : 3 : 5-Tribromopyridine.

Needles from EtOH.Aq. M.p. 46°. Sol. EtOH, Et_2O, C_6H_6. Volatile in steam. Feebly basic.

$B_2,HgCl_2$: cryst. from EtOH.Aq. M.p. 181–2°.

Fischer, Chur, *J. prakt. Chem.*, 1916, **93**, 372.
Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

2 : 3 : 6-Tribromopyridine.

M.p. 82°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

2 : 4 : 5-Tribromopyridine.

Cryst. from EtOH.Aq. M.p. 66·5–67·5°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

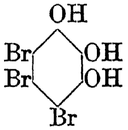
2 : 4 : 6-Tribromopyridine.
Leaflets from EtOH. M.p. 107°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 946.

3 : 4 : 5-Tribromopyridine.
Cryst. from EtOH. M.p. 106–7°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 950.

Tribromopyrogallol



$C_6H_3O_3Br_3$ MW, 363

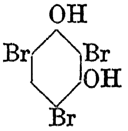
Reddish or brownish needles or leaflets + $1H_2O$ from EtOH.Aq. M.p. 168–70°. Very sol. EtOH. Sol. hot H_2O.

Tri-Me ether : $C_9H_9O_3Br_3$. MW, 405. Cryst. M.p. 81·5°.

Tri-Et ether : $C_{12}H_{15}O_3Br_3$. MW, 447. M.p. 38–9°.

Moore, Thomas, *J. Am. Chem. Soc.*, 1917, **39**, 987.

2 : 4 : 6-Tribromoresorcinol



$C_6H_3O_2Br_3$ MW, 347

Needles from H_2O. M.p. 112°. Sol. EtOH. Spar. sol. cold H_2O. Gives reactions of quinones.

Me ether : $C_7H_5O_2Br_3$. MW, 361. Needles. M.p. 104° (99°). Sol. EtOH, Et_2O, C_6H_6, ligroin.

Di-Me ether : $C_8H_7O_2Br_3$. MW, 375. Cryst. from EtOH.Aq. M.p. 68–9°. Sol. EtOH, Et_2O, C_6H_6, ligroin. Insol. H_2O.

Me-Et ether : $C_9H_9O_2Br_3$. MW, 389. Needles from EtOH.Aq. M.p. 75°.

Di-Et ether : $C_{10}H_{11}O_2Br_3$. MW, 403. M.p. 68–9°.

Monoacetyl : cryst. from CS_2. M.p. 114°.

Diacetyl : needles. M.p. 108°.

Monobenzoyl : prisms from $CHCl_3$–pet. ether. M.p. 120°.

Benedikt, *Monatsh.*, 1883, **4**, 227.
Raiford, Heyl, *Am. Chem. J.*, 1910, **44**, 215.

Tribromosuccinic Acid

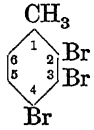
$CBr_2·COOH$
$CHBr·COOH$

$C_4H_3O_4Br_3$ MW, 355

Plates from Et_2O–C_6H_6. M.p. 136°. Very sol. H_2O, EtOH, Et_2O. Mod. sol. C_6H_6. Very spar. sol. CS_2, ligroin. Hot H_2O ⟶ 2 : 2-dibromoacrylic acid.

Lossen, Bergau, *Ann.*, 1906, **348**, 265.

2 : 3 : 4-Tribromotoluene



$C_7H_5Br_3$ MW, 329

Cryst. from AcOH. M.p. 45–6°. D^{20} 2·456.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 510.

2 : 3 : 5-Tribromotoluene.
Cryst. from Et_2O–toluene. M.p. 53–4°. D^{17} 2·467.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 511, 520.

2 : 3 : 6-Tribromotoluene.
Leaflets from ligroin or $CHCl_3$. M.p. 58–9° (60·5°). D^{17} 2·471.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 513, 519.

2 : 4 : 5-Tribromotoluene.
Needles from EtOH. M.p. 112–13°. D^{17} 2·472.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515, 520.

2 : 4 : 6-Tribromotoluene.
Needles from Et_2O–AcOEt. M.p. 70° (65–6°). D^{17} 2·479. Very spar. sol. EtOH.

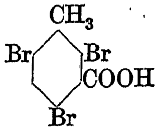
Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515.

3 : 4 : 5-Tribromotoluene.
Needles from Et_2O–EtOH. M.p. 91° (88–9°). D^{17} 2·429.

Asinger, *J. prakt. Chem.*, 1935, **142**, 297.

4 : ω : ω-Tribromotoluene.
See p-Bromobenzylidene bromide.

2 : 4 : 6-Tribromo-*m*-toluic Acid



$C_8H_5O_2Br_3$ MW, 373

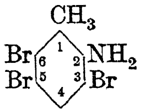
Needles from 50% MeOH. M.p. 187–188·5° after sintering at 160°.

Amide : $C_8H_6ONBr_3$. MW, 372. Cryst. from 50% EtOH. M.p. 199–200°.

Nitrile : $C_8H_4NBr_3$. MW, 354. Cryst. from pet. ether or cyclohexane. M.p. 122–3°.

Weissberger, Bach, Strasser, *J. Chem. Soc.*, 1935, 70.

3 : 5 : 6-Tribromo-*o*-toluidine



$C_7H_6NBr_3$ MW, 344

M.p. 87°. Sol. Et_2O, C_6H_6, hot EtOH. Mod. sol. pet. ether.

N-Acetyl : 3 : 5 : 6-tribromoacet-*o*-toluidide. M.p. 218°.

Blanksma, *Chem. Zentr.*, 1914, I, 971.

2 : 4 : 6-Tribromo-*m*-toluidine.

Needles from EtOH. M.p. 101°. Spar. sol. EtOH.

N-Acetyl : 2 : 4 : 6-tribromoacet-*m*-toluidide. Cryst. from EtOH. M.p. 211·5–212°.

N-Diacetyl : m.p. 103°.

N-Chloroacetyl : m.p. 196°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 515.
Fuchs, *Monatsh.*, 1915, **36**, 132.
Bureš, Balada, *Chem. Zentr.*, 1927, II, 1345.
Dains *et al.*, *Chem. Abstracts*, 1938, **32**, 3397.
Qvist, *Chem. Abstracts*, 1947, **41**, 2397.

2 : 5 : 6-Tribromo-*m*-toluidine.

M.p. 93–4°.

N-Acetyl : 2 : 5 : 6-tribromoacet-*m*-toluidide. M.p. 179–81°.

Nevile, Winther, *Ber.*, 1880, **13**, 974.

4 : 5 : 6-Tribromo-*m*-toluidine.

M.p. 96–96·8°.

N-Acetyl : 4 : 5 : 6-tribromoacet-*m*-toluidide. Needles from EtOH. M.p. 171–3°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 510.

2 : 3 : 5-Tribromo-*p*-toluidine.

Needles from EtOH. M.p. 82·5–83°.

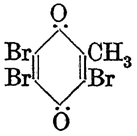
Nevile, Winther, *Ber.*, 1881, **14**, 418.

2 : 3 : 6-Tribromo-*p*-toluidine.

Needles from EtOH. M.p. 118°. Volatile in steam.

Cohen, Dutt, *J. Chem. Soc.*, 1914, **105**, 514.

3 : 5 : 6-Tribromotoluquinone (3 : 5 : 6-*Tribromo-2-methylbenzoquinone*)



$C_7H_3O_2Br_3$ MW, 359

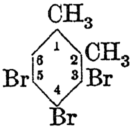
Yellow plates from EtOH. M.p. 235–6°. Sol. Et_2O, C_6H_6. Very spar. sol. cold EtOH. Insol. H_2O.

Fichter, Rinderspacher, *Helv. Chim. Acta*, 1927, **10**, 41.
Zincke, Klostermann, *Ber.*, 1907, **40**, 679.

Tribromoveratrol.

See under Tribromocatechol.

3 : 4 : 5-Tribromo-*o*-xylene



$C_8H_7Br_3$ MW, 343

Needles from Me_2CO. M.p. 105°.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 354.

3 : 4 : 6-Tribromo-*o*-xylene.

Needles. M.p. 86°. Sol. Et_2O, C_6H_6. Spar. sol. cold EtOH, cold Me_2CO.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 354.

2 : 4 : 5-Tribromo-*m*-xylene.

Cryst. from EtOH. M.p. 87°.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 361.

2 : 4 : 6-Tribromo-*m*-xylene.

Prisms from Et_2O–C_6H_6. M.p. 85°.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 357.

4 : 5 : 6-Tribromo-*m*-xylene.

Cryst. M.p. 105°.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 357.

2 : 3 : 5-Tribromo-*p*-xylene.

M.p. 89°. Sol. Et_2O. Spar. sol. Me_2CO, cold EtOH.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, **25**, 362.

Tributylamine

$(CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2)_3N$

$C_{12}H_{27}N$ MW, 185

B.p. 216·5°. D_{20}^{20} 0·7782.

B,HFeCl₄ : yellow needles. M.p. 171°.

Butyl iodide : tetrabutylammonium iodide. M.p. 144–5°. Sol. H_2O, EtOH.

Picrate : m.p. 105·6°.

Butyl picrate : tetrabutylammonium picrate. M.p. 90·3°.

Benzyl chloride : tributylbenzylammonium chloride. M.p. 185°.

I.G., F.P., 685,345, (*Chem. Abstracts*, 1930, **24**, 5765).
Skita, Keil, *Monatsh.*, 1929, **53** and **54**, 759.
Lieben, Rossi, *Ann.*, 1871, **158**. 172.
Borrows *et al.*, *J. Chem. Soc.*, 1947, 197.

Tributylbenzylammonium chloride.

See under Tributylamine.

Tributyl phosphate.
See under Phosphoric Acid.

Tributyrin (*Glycerol tri*-n-*butyrate*)

$CH_2·O·CO·CH_2·CH_2·CH_3$
$CH·O·CO·CH_2·CH_2·CH_3$
$CH_2·O·CO·CH_2·CH_2·CH_3$

$C_{15}H_{26}O_6$ MW, 302

Colourless liq. with bitter taste. B.p. 305–10° (287–8°), 190°/15 mm. n_D^{20} 1·43587. D^{21} 1·027.

Weatherby, McIlvaine, Matlin, *J. Am. Chem. Soc.*, 1925, **47**, 2249.

Tricaprin.
See under n-Capric Acid.

Tricaproin.
See under n-Caproic Acid.

Tricaprylin.
See under n-Caprylic Acid.

Tricarballylic Acid (*Propane*-1 : 2 : 3-*tricarboxylic acid*)

$CH_2·COOH$ α
$CH·COOH$ β
$CH_2·COOH$

$C_6H_8O_6$ MW, 176

Prisms from H_2O or Et_2O. M.p. 166°. Sol. H_2O, EtOH. Mod. sol. Et_2O. $k = 2·2 \times 10^{-4}$ at 25°.

α-Me ester: $C_7H_{10}O_6$. MW, 190. Oil. $k = 7·5 \times 10^{-5}$ at 25°.

β-Me ester: oil. $k = 9·35 \times 10^{-5}$ at 25°.

Tri-Me ester: $C_9H_{14}O_6$. MW, 218. B.p. 205–8°/48 mm., 150°/13 mm. D_4^{20} 1·18221. n_D 1·4398.

Tri-Et ester: $C_{12}H_{20}O_6$. MW, 260. Oil. B.p. 295–305°, 180–5°/20 mm. Spar. sol. H_2O.

Triallyl ester: $C_{15}H_{20}O_6$. MW, 296. B.p. 215°/24 mm. $D_4^{18·4}$ 1·0953. $n_D^{15·9}$ 1·46747.

Trichloride: $C_6H_5O_3Cl_3$. MW, 231·5. Liq. B.p. 140°/14 mm.

Triamide: $C_6H_{11}O_3N_3$. MW, 173. Prisms from H_2O. M.p. 205–7° decomp. Sol. H_2O. Insol. EtOH, Et_2O, $CHCl_3$.

Trinitrile: 1 : 2 : 3-tricyanopropane. $C_6H_5N_3$. MW, 119. Needles from Et_2O. M.p. 47°. Sol. hot H_2O. Spar. sol. Et_2O, cold EtOH.

Trihydrazide: cryst. from EtOH.Aq. M.p. 195–6°. *B,3HCl*: m.p. 148° decomp.

Trianilide: needles from $PhNO_2$. M.p. 252°.

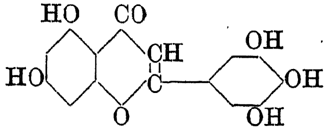
αβ-Anhydride: $C_6H_6O_5$. MW, 158. Needles from AcOH–$CHCl_3$. M.p. 131–2°. B.p. 215–25°/45 mm. Sol. H_2O, EtOH, AcOH. Spar. sol. Et_2O, $CHCl_3$.

αβ-Imide: $C_6H_7O_4N$. MW, 157. Prisms from C_6H_6. M.p. 127–8°. Sol. H_2O, EtOH. Spar. sol. Et_2O, C_6H_6. *Amide*: cryst. from EtOH.Aq. M.p. 173°.

αβ-Anil: $C_{12}H_{11}O_4N$. MW, 233. Plates from H_2O. M.p. 137°. *Me ester*: $C_{13}H_{13}O_4N$. MW, 247. Needles from Et_2O–EtOH. M.p. 106°. *Et ester*: $C_{14}H_{15}O_4N$. MW, 261. Needles. M.p. 90°. *Anilide*: needles from EtOH.Aq. M.p. 168°.

Clarke, Murray, *Organic Syntheses*, Collective Vol. I, 508.
Bertram, *Ber.*, 1905, **38**, 1620.
Bone, Sprankling, *J. Chem. Soc.*, 1902, **81**, 34.
Emery, *Ber.*, 1891, **24**, 597; 1889, **22**, 2920.
Andersen, U.S.P. 2,118,033, (*Chem. Abstracts*, 1938, **32**, 5584).

Tricetin (5 : 7 : 3′ : 4′ : 5′-*Pentahydroxy-flavone*)



$C_{15}H_{10}O_7$ MW, 302

Needles + H_2O from EtOH.Aq. Decomp. above 330°. Conc. H_2SO_4 ⟶ yellow sol. NaOH ⟶ orange-red sol. Alc. $FeCl_3$ ⟶ reddish-brown col. ⟶ olive-green.

3′ : 5′-*Di-Me ether*: *see* Tricin.

3′ : 4′ : 5′-*Tri-Me ether*: $C_{18}H_{16}O_7$. MW, 344. Yellow needles from AcOH. M.p. 269–70°. Conc. H_2SO_4 and NaOH give yellow sols. Alc. $FeCl_3$ ⟶ reddish-brown to olive-green col. *Diacetyl*: needles from EtOH. M.p. 160–2°.

Penta-Me ether: $C_{20}H_{20}O_7$. MW, 372. Needles from MeOH. M.p. 192–3°.

Tetra-acetyl: needles. M.p. 260–1°.

Penta-acetyl: needles from EtOH–AcOH. M.p. 244° (241–2°).

Anderson, *Chem. Zentr.*, 1932, II, 3899; 1933, II, 2012.

Tricetylamine.
See under Cetylamine.

Trichloroacetaldehyde.
See Chloral.

Trichloroacetanilide.
See under Trichloroaniline.

Trichloroacetic Acid

$CCl_3·COOH$

$C_2HO_2Cl_3$ MW, 163·5

Deliquescent cryst. M.p. 58°. B.p. 196·5°, 141–2°/25 mm. D_{70}^{70} 1·6237. Sol. H_2O. $k = 1·2$ approx. Hot H_2O or dil. alkalis ⟶ chloroform + CO_2. Conc. alkalis ⟶ formic acid.

Me ester: $C_3H_3O_2Cl_3$. MW, 177·5. B.p. 152°, 52–4°/12 mm.

Et ester: $C_4H_5O_2Cl_3$. MW, 191·5. B.p. 167·1°/754·8 mm., 60–1°/12 mm. D_{15}^{1} 1·3886. n_D^{20} 1·45068.

Propyl ester: $C_5H_7O_2Cl_3$. MW, 205·5. B.p. 187°.

Isobutyl ester: $C_6H_9O_2Cl_3$. MW, 219·5. B.p. 187–9°.

tert.-*Butyl ester*: m.p. 25·5°. B.p. 37°/1 mm. D_4^{25} 1·2363. n^{25} 1·4398.

active-*Amyl ester*: $C_7H_{11}O_2Cl_3$. MW, 233·5. B.p. 210–12°/721 mm. D_4^{22} 1·233. $n_D^{21\cdot4}$ 1·4517. $[\alpha]_D^{22}$ + 3·54°.

Neopentyl ester: b.p. 202°/760 mm. D_4^{20} 1·2142. n_D^{20} 1·4373.

Allyl ester: $C_5H_5O_2Cl_3$. MW, 203·5. B.p. 183–4°/766 mm.

Vinyl ester: $C_4H_3O_2Cl_3$. MW, 189·5. B.p. 149°.

Cyclohexyl ester: b.p. 105–7°/3 mm., 85–8°/2 mm. D_{15}^{15} 1·2864. n_D^{15} 1·4840.

Phenyl ester: $C_8H_5O_2Cl_3$. MW, 239·5. B.p. 254–5° decomp.

2-*Naphthyl ester*: m.p. 86–7°.

Benzyl ester: viscous oil. B.p. 178·5°/50 mm. D_4^4 1·3887. $n_D^{18\cdot8}$ 1·5288.

Benzhydryl ester: m.p. 47–8°.

Anhydride: $C_4O_3Cl_6$. MW, 309. B.p. 222–4° decomp., 140°/110 mm. D^{20} 1·6908.

Chloride: C_2OCl_4. MW, 182. B.p. 118°. D_4^0 1·6564.

Bromide: C_2OCl_3Br. MW, 226·5. B.p. 143°. D_{15}^{15} 1·900.

Iodide: C_2OCl_3I. MW, 273·5. B.p. about 180°.

Amide: $C_2H_2ONCl_3$. MW, 162·5. Cryst. from H_2O. M.p. 141°. B.p. 238–9°/746 mm. Sol. EtOH, Et_2O. Spar. sol. H_2O. Sublimes in plates.

Methylamide: cryst. from Et_2O. M.p. 105–6°.

Dimethylamide: b.p. 230–3° slight decomp. D^{15} 1·441.

Ethylamide: cryst. M.p. 74°. B.p. 229–30° slight decomp.

Diethylamide: prisms with odour of peppermint. M.p. 27°.

Nitrile: C_2NCl_3. MW, 144·5. B.p. 83–4°. $D^{12\cdot2}$ 1·439.

Imide: di-trichloroacetamide. $C_4HO_2NCl_6$. MW, 308. Cryst. from pet. ether. M.p. 86°.

Anilide: cryst. from EtOH.Aq. M.p. 95–7°.

o-*Nitroanilide*: yellow needles from EtOH. M.p. 70–2°.

p-*Nitroanilide*: needles from EtOH. M.p. 146–7°.

Phenylhydrazide: m.p. 123°.

Anschütz, Haslam, *Ann.*, 1889, **253**, 124.
Judson, *Ber.*, 1870, **3**, 782.
Clermont, *Ann. chim. phys.*, 1885, **6**, 135.
Quayle, Morton, *J. Am. Chem. Soc.*, 1940, **62**, 1170.
Dewey, Shaskey, *J. Am. Chem. Soc.*, 1941, **63**, 3526.
Stempel, Schaffel, *J. Am. Chem. Soc.*, 1942, **64**, 470.
Zavgorodniĭ, *J. Gen. Chem. U.S.S.R.*, 1944, **14**, 270.
Scovill, Burk, Lankelma, *J. Am. Chem. Soc.*, 1944, **66**, 1039.
Hardegger, Heweihi, Robinet, *Helv. Chim. Acta*, 1948, **31**, 439.

Trichloro-acetoisopropyl Alcohol.

See Chloralacetone.

1 : 1 : 1-Trichloroacetone

$CH_3·CO·CCl_3$

$C_3H_3OCl_3$ MW, 161·5

Liq. with odour of camphor. B.p. 149°/764 mm. (134°), 57°/48 mm.

Semicarbazone: needles. M.p. 140° decomp.

Blaise, *Compt. rend.*, 1912, **155**, 1253.
Schlotterbeck, *Ber.*, 1909, **42**, 2561.

1 : 1 : 3-Trichloroacetone

$CH_2Cl·CO·CHCl_2$

$C_3H_3OCl_3$ MW, 161·5

B.p. 172°.

Cloëz, *Ann. chim. phys.*, 1886, **9**, 176.

Trichloro-acet-toluidide.

See under Trichlorotoluidine.

Trichloroacrylic Acid

$CCl_2{:}CCl·COOH$

$C_3HO_2Cl_3$ MW, 175·5

Prisms from CS_2. M.p. 76°. Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O. Spar. sol. cold H_2O. k = about $5·4 \times 10^{-2}$ at 25°.

Et ester: $C_5H_5O_2Cl_3$. MW, 203·5. Liq. with odour of peppermint. B.p. 192–4°, 112–14°/50 mm. D_4^{20} 1·3740. n_D^{20} 1·4839.

Anhydride: $C_6O_3Cl_6$. MW, 333. Cryst. M.p. 39–40°. Insol. H_2O.

Chloride: C_3OCl_4. MW, 194. B.p. 158°. $n_D^{18\cdot5}$ 1·52709.

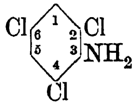
Amide: $C_3H_2ONCl_3$. MW, 174·5. Needles. M.p. 87°. Sol. EtOH, Et_2O, hot H_2O. Spar. sol. cold H_2O.

Anilide: needles from pet. ether. M.p. 98°.

Nitrile: C_3NCl_3. MW, 156·5. Cryst. M.p. 20°. $n_D^{20\cdot5}$ 1·5100.

Prins, D.R.P., 261,689, (*Chem. Zentr.*, 1913, II, 394).
Fritsch, *Ann.*, 1897, **297**, 315.
Mabery, *Am. Chem. J.*, 1887, **9**, 3.
Bergmann, Haskelberg, *J. Am. Chem. Soc.*, 1941, **63**, 1437.

2 : 4 : 6-Trichloro-*m*-aminophenol



$C_6H_4ONCl_3$ MW, 212·5

Leaflets from ligroin. M.p. 95–6°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O. Aq. sol. ⟶ violet-red col. with $FeCl_3$.

N-*Acetyl*: needles from toluene. M.p. 185–186·5°. $FeCl_3$ ⟶ violet col.

Jacobs, Heidelberger, Rolf, *J. Am. Chem. Soc.*, 1919, **41**, 463.
Daccomo, *Ber.*, 1885, **18**, 1166.

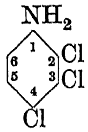
2 : 3 : 5-Trichloro-*p*-aminophenol.
M.p. 166–7°.
Me ether : m.p. 88·5–89°.

de Traz, *Helv. Chim. Acta*, 1947, **30**, 232.

2 : 3 : 6-Trichloro-*p*-aminophenol.
Needles from EtOH. M.p. 159° ⟶ a brown liq. Sol. EtOH, Et_2O, hot H_2O.
N-2 : 4-*Dinitrophenyl* : orange-red cryst. M.p. 211°. *Acetyl* : yellow prisms. M.p. 153°.

Kohn, Fink, *Monatsh.*, 1930, **56**, 138.
Schmitt, Andresen, *J. prakt. Chem.*, 1881, **24**, 426.

2 : 3 : 4-Trichloroaniline



$C_6H_4NCl_3$ MW, 196·5

Needles from ligroin. M.p. 67·5°. B.p. 292°/774 mm. Sol. EtOH.
N - *Acetyl* : 2 : 3 : 4 - trichloroacetanilide. Needles. M.p. 123°.

Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 56.
Beilstein, Kurbatow, *Ann.*, 1879, **196**, 233.

2 : 3 : 5-Trichloroaniline.
Needles from pet. ether. M.p. 73°.

Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2921.

2 : 3 : 6-Trichloroaniline.
Cryst. from EtOH. M.p. 63–4°.
N-*Acetyl* : 2 : 3 : 6-trichloroacetanilide. Cryst. from EtOH.Aq. M.p. 172–3° (134·5°).

Hüffer, *Rec. trav. chim.*, 1921, **40**, 457.
Chattaway, Orton, Hurtley, *J. Chem. Soc.*, 1900, **77**, 802.

2 : 4 : 5-Trichloroaniline.
Needles from ligroin. M.p. 96·5°. B.p. about 270°. Sol. EtOH, CS_2. Spar. sol. ligroin. Volatile in steam.
N-*Acetyl* : 2:4:5-trichloroacetanilide. Needles from C_6H_6. M.p. 190°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3044.
Beilstein, Kurbatow, *Ann.*, 1879, **196**, 233.

2 : 4 : 6-Trichloroaniline.
Needles from ligroin. M.p. 78·5°. B.p. 262°/746 mm. Sol. EtOH, Et_2O, CS_2, ligroin.
N - *Me* : 2 : 4 : 6 - trichloromethylaniline. $C_7H_6NCl_3$. MW, 210·5. Needles. M.p. 32°. B.p. 260°. N-*Acetyl* : m.p. 89–90°. N-*Benzoyl* : m.p. 96–7°. N-*Nitroso* : m.p. 66·5–67°.
N-*Di-Me* : 2 : 4 : 6-trichlorodimethylaniline. $C_8H_8NCl_3$. MW, 224·5. B.p. 247°/745 mm., 128–38°/20 mm. n_D^{20} 1·5649.
N-*Et* : 2:4:6-trichloroethylaniline. $C_8H_8NCl_3$. MW, 224·5. B.p. 148–53°/25 mm. N-*Acetyl* : m.p. 50–1°. N-*Benzoyl* : m.p. 127–8°.
N - *Formyl* : 2 : 4 : 6 - trichloroformanilide. Needles from EtOH. M.p. 180°.
N - *Acetyl* : 2 : 4 : 6 - trichloroacetanilide. Needles. M.p. 207–8° (204°).
N-*Diacetyl* : prisms from EtOH. M.p. 81–2°.
N-*Propionyl* : prisms from EtOH. M.p. 161°.
N-*Benzoyl* : needles from EtOH. M.p. 174°.
Picrate : crimson prisms from EtOH. M.p. 82·5°.

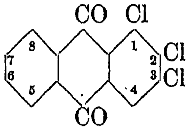
Chattaway, Irving, *J. Chem. Soc.*, 1933, 142.
Beilstein, Kurbatow, *Ann.*, 1879, **196**, 230.
Emerson, *J. Am. Chem. Soc.*, 1941, **63**, 2023.

3 : 4 : 5-Trichloroaniline.
Needles from EtOH.Aq. M.p. 100° (89°).
N-*Acetyl* : 3 : 4 : 5-trichloroacetanilide. M.p. 207–8°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3043.
Holleman, *Rec. trav. chim.*, 1918, **37**, 196.

Trichloroanisole.
See under Trichlorophenol.

1 : 2 : 3-Trichloroanthraquinone



$C_{14}H_5O_2Cl_3$ MW, 311·5

Yellow needles from AcOH. M.p. 194–5°.

Goldberg, *J. Chem. Soc.*, 1931, 1789.

1 : 2 : 4-Trichloroanthraquinone.
Yellow cryst. from $CHCl_3$. M.p. 185·5°. Sol. $CHCl_3$, C_6H_6. Mod. sol. Et_2O, AcOH. Spar. sol. EtOH.

Graebe, Rostowzew, *Ber.*, 1901, **34**, 2113.
Goldberg, *J. Chem. Soc.*, 1931, 1771.

1 : 2 : 5-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 235–6°.

Goldberg, *J. Chem. Soc.*, 1931, 1792.
Cf. Schilling, *Ber.*, 1913, **46**, 1068.

1 : 2 : 6-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 222–3°.

Goldberg, *J. Chem. Soc.*, 1931, 1784.

1 : 2 : 7-Trichloroanthraquinone.
Needles from AcOH. M.p. 225–6° (259–60°).

Goldberg, *J. Chem. Soc.*, 1931, 1786.
Keimatsu, Hirano, Tanabe, *Chem. Abstracts*, 1929, **23**, 4214.

1 : 3 : 6-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 212–13°.

Goldberg, *J. Chem. Soc.*, 1931, 1790.

1 : 3 : 7-Trichloroanthraquinone.
Lemon-yellow needles from AcOH. M.p. 216–17°.

Goldberg, *J. Chem. Soc.*, 1931, 1790.

1 : 4 : 5-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 258° (253–4°).

Badische, D.R.P., 214,714, (*Chem. Zentr.*, 1909, II, 1603).
Goldberg, *J. Chem. Soc.*, 1931, 1791.
Cf. Schilling, *Ber.*, 1913, **46**, 1068.

1 : 4 : 6-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 238°.

Goldberg, *J. Chem. Soc.*, 1931, 1784.
Jaroschy, *Monatsh.*, 1913, **34**, 3.

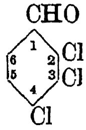
2 : 3 : 5-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 227–8°.

Goldberg, *J. Chem. Soc.*, 1932, 80.

2 : 3 : 6-Trichloroanthraquinone.
Yellow needles from AcOH. M.p. 245°.

Goldberg, *J. Chem. Soc.*, 1932, 81.

2 : 3 : 4-Trichlorobenzaldehyde



$C_7H_3OCl_3$ MW, 209·5

Needles from EtOH. M.p. 90°.

Seelig, *Ann.*, 1887, **237**, 149.

2 : 3 : 5-Trichlorobenzaldehyde.
Needles from EtOH. M.p. 56°. Sol. most org. solvents. Volatile in steam. $KMnO_4 \longrightarrow$ 2 : 3 : 5-trichlorobenzoic acid.

Hodgson, Beard, *J. Chem. Soc.*, 1927, 2382.

2 : 3 : 6-Trichlorobenzaldehyde.
Needles from ligroin. M.p. 86–7°.

Geigy, D.R.P., 199,943, (*Chem. Zentr.*, 1908, II, 363).

2 : 4 : 5-Trichlorobenzaldehyde.
Needles. M.p. 112–13°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Volatile in steam. Oxidises in air.

Seelig, *Ann.*, 1887, **237**, 148.
Beilstein, Kuhlberg, *Ann.*, 1869, **152**, 238.

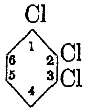
2 : 4 : 6-Trichlorobenzaldehyde.
Needles from ligroin. M.p. 58–9°.

Geigy, D.R.P., 199,943, (*Chem. Zentr.*, 1908, II, 363).

3 : 4 : 5-Trichlorobenzaldehyde.
Needles from EtOH.Aq. M.p. 90–1°. $KMnO_4 \longrightarrow$ 3 : 4 : 5-trichlorobenzoic acid.
Phenylhydrazone : yellow cryst. M.p. 147°.
p-*Nitrophenylhydrazone* : orange needles from $PhNO_2$. M.p. 342°.
Semicarbazone : cryst. from EtOH. M.p. 252–4°.

van der Bunt, *Rec. trav. chim.*, 1929, **48**, 131.

1 : 2 : 3-Trichlorobenzene



$C_6H_3Cl_3$ MW, 181·5

Plates from EtOH. M.p. 53–4°. B.p. 218–19°. Mod. sol. EtOH. Volatile in steam.

Holleman, *Rec. trav. chim.*, 1918, **37**, 196.
Beilstein, Kurbatow, *Ann.*, 1878, **192**, 234.

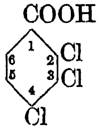
1 : 2 : 4-Trichlorobenzene.
M.p. 17°. B.p. 210°. D^{26} 1·4460.

van de Lande, *Rec. trav. chim.*, 1932, **51**, 98.
Cohen, Hartley, *J. Chem. Soc.*, 1905, **87**, 1363.

1 : 3 : 5-Trichlorobenzene.
Needles. M.p. 63·5°. B.p. 208·5°/763·8 mm.

Holleman, *Rec. trav. chim.*, 1918, **37**, 198.
Jackson, Lamai, *Am. Chem. J.*, 1896, **18**, 667.

2 : 3 : 4-Trichlorobenzoic Acid



$C_7H_3O_2Cl_3$ MW, 225·5

Needles from H_2O. M.p. 186–7°.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1328.

2 : 3 : 5-Trichlorobenzoic Acid.
Needles from H_2O. M.p. 163°. Sol. common org. solvents. Spar. sol. H_2O. Non-volatile in steam.
Chloride : $C_7H_2OCl_4$. MW, 244. Cryst. from AcOEt. M.p. 36°.
Amide : $C_7H_4ONCl_3$. MW, 224·5. Needles from AcOH.Aq. M.p. 204–5°.
Nitrile : $C_7H_2NCl_3$. MW, 206·5. Needles from EtOH.Aq. M.p. 87°. Volatile in steam.

Hodgson, Beard, *J. Chem. Soc.*, 1927, 2382.
Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1331.
Matthews, *J. Chem. Soc.*, 1901, **79**, 46.

2 : 4 : 5-Trichlorobenzoic Acid.
Needles from H_2O. M.p. 168°. Sol. EtOH. Prac. insol. cold H_2O. Sublimes in needles.
Et ester : $C_9H_7O_2Cl_3$. MW, 253·5. Needles from EtOH. M.p. 65°. Mod. sol. EtOH.

Chloride: m.p. 28°. B.p. 272° slight decomp., 125°/2 mm. Sol. Et_2O, CS_2, C_6H_6.
Amide: needles from C_6H_6. M.p. 167·5°. Sol. EtOH. Spar. sol. hot H_2O. Very spar. sol. Et_2O, CS_2.
Nitrile: m.p. 104°.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1335.
Beilstein, Kuhlberg, *Ann.*, 1869, **152**, 234.
Baker, Tweed, *J. Chem. Soc.*, 1941, 796.

2 : 4 : 6-Trichlorobenzoic Acid.

Cryst. from H_2O. M.p. 164°. Sol. EtOH, Et_2O, $CHCl_3$.
Chloride: oil. B.p. 275°.
Amide: cryst. M.p. 181°. Mod. sol. hot H_2O.
Nitrile: needles. M.p. 77·5°. Sol. EtOH, Et_2O. Spar. sol. hot H_2O.

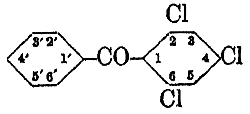
Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1336.
Sudborough, *J. Chem. Soc.*, 1895, **67**, 602.

3 : 4 : 5-Trichlorobenzoic Acid.

Needles from EtOH.Aq. M.p. 210°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. CS_2. Prac. insol. H_2O. Sublimes in needles. Spar. volatile in steam.
Et ester: needles. M.p. 86°.
Chloride: prisms. M.p. 36°. Sol. Et_2O, CS_2, C_6H_6.
Amide: needles from C_6H_6. M.p. 176°. Sol. EtOH, Et_2O. Insol. H_2O.

van der Bunt, *Rec. trav. chim.*, 1929, **48**, 132.
Salkowski, *Ann.*, 1872, **163**, 28.

2 : 4 : 6-Trichlorobenzophenone



$C_{13}H_7OCl_3$ MW, 285·5

Cryst. from pet. ether. M.p. 130·5°. B.p. 356°/763 mm.

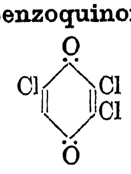
Montagne, *Rec. trav. chim.*, 1907, **26**, 274.

2 : 5 : 2′-Trichlorobenzophenone.

Needles from EtOH. M.p. 145–7°.

Ganzmüller, *J. prakt. Chem.*, 1933, **138**, 311.

Trichloro-*p*-benzoquinone



$C_6HO_2Cl_3$ MW, 211·5

Yellow plates from EtOH. M.p. 169–70°. Sol. Et_2O, hot EtOH. Insol. cold H_2O. Sublimes.

Erdélyi, *Ber.*, 1930, **63**, 1200.
v. Knapp, Schultz, *Ann.*, 1881, **210**, 174.

Trichlorobromomethane (*Trichloromethyl bromide*)

CCl_3Br

CCl_3Br MW, 198·5

M.p. −21°. B.p. 104·3°. D_4^0 2·05496. Photochem. reaction with Cl ⟶ carbon tetrachloride. Sensitive to light.

Wonters, *Bull. sci. acad. roy. Belg.*, 1934, **20**, 782.
Löw, *Z. Chem.*, 1869, 624.

1 : 1 : 3-Trichlorobutane

$CH_3{\cdot}CHCl{\cdot}CH_2{\cdot}CHCl_2$

$C_4H_7Cl_3$ MW, 161·5

B.p. 152°/753 mm. D^{15} 1·317. n_D^{15} 1·46.

Dulou, Bulteau, *Bull. soc. chim.*, 1947, 924.

1 : 2 : 3-Trichlorobutane

$CH_3{\cdot}CHCl{\cdot}CHCl{\cdot}CH_2Cl$

$C_4H_7Cl_3$ MW, 161·5

B.p. 165–8°/725 mm., 79–80°/32 mm., 62–3°/28 mm. D^0 1·3241, D_4^{20} 1·3164. n_C^{18} 1·4745, n_D^{20} 1·479. Sol. EtOH, Et_2O, $CHCl_3$. Insol. H_2O.

de Montmollin, Matile, *Helv. Chim. Acta*, 1924, **7**, 106.
Charon, *Ann. chim. phys.*, 1899, **17**, 230.
Tischchenko, *J. Gen. Chem. U.S.S.R.*, 1936, **6**, 1549.

2 : 2 : 3-Trichlorobutane

$CH_3{\cdot}CHCl{\cdot}CCl_2{\cdot}CH_3$

$C_4H_7Cl_3$ MW, 161·5

B.p. 143–5°. D_4^{18} 1·263. n_C^{18} 1·4637.

Tischchenko, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 1232.

Note.—Losanitsch, *Chem. Zentr.*, 1913, II, 754, described a trichlorobutane of unknown constitution, b.p. 90°/16 mm.

2 : 2 : 3-Trichloro-*n*-butyl Alcohol

$CH_3{\cdot}CHCl{\cdot}CCl_2{\cdot}CH_2OH$

$C_4H_7OCl_3$ MW, 177·5

Prisms. M.p. 61·5–62°. B.p. 199–200°, 120°/45 mm. Sol. EtOH, Et_2O. Spar. sol. H_2O. Reduces warm Fehling's. HNO_3 ⟶ 1 : 1 : 2-trichlorobutyric acid.
Phosphate: needles from EtOH. M.p. 85·3°.

Mering, *Z. physiol. Chem.*, 1882, **6**, 493.
Garzarolli-Thurnlakh, Popper, *Ann.*, 1884, **223**, 166.
Floutz, *J. Am. Chem. Soc.*, 1946, **68**, 2490.

1 : 1 : 1-Trichloro-*tert*.-butyl Alcohol.

See Chloretone.

1 : 1 : 2-Trichlorobutyraldehyde.

See Butylchloral.

1 : 1 : 3-Trichlorobutyraldehyde

$CH_2Cl{\cdot}CH_2{\cdot}CCl_2{\cdot}CHO$

$C_4H_5OCl_3$ MW, 175·5

Liq. Spar. sol. H_2O. Forms no hydrate. Oxidises slowly in air. $HNO_3 \longrightarrow$ 1 : 1 : 3-trichlorobutyric acid. Forms bisulphite comp.

Natterer, *Monatsh.*, 1883, **4**, 551.

1 : 1 : 2-Trichlorobutyric Acid

$CH_3{\cdot}CHCl{\cdot}CCl_2{\cdot}COOH$

$C_4H_5O_2Cl_3$ MW, 191·5

Plates or needles from pet. ether. M.p. 60°. B.p. 236–8°. k = about 0·18 at 18°.

Et ester: $C_6H_9O_2Cl_3$. MW, 219·5. B.p. 212°, 101·5°/17 mm. D_{20}^{20} 1·3138.

Chloride: $C_4H_4OCl_4$. MW, 210. B.p. 162–6°.

Amide: $C_4H_6ONCl_3$. MW, 190·5. Plates from EtOH. M.p. 96°. Spar. sol. H_2O.

Krämer, Pinner, *Ber.*, 1870, **3**, 389.
Valentin, *Ber.*, 1895, **28**, 2661.
Plump, U.S.P. 2,370,577, (*Chem. Abstracts*, 1945, **39**, 4085); U.S.P. 2,443,118, (*Chem. Abstracts*, 1948, **42**, 7320).

1 : 1 : 3-Trichlorobutyric Acid

$CH_2Cl{\cdot}CH_2{\cdot}CCl_2{\cdot}COOH$

$C_4H_5O_2Cl_3$ MW, 191·5

Cryst. M.p. 73–5°. Sol. 20 parts H_2O.

Et ester: $C_6H_9O_2Cl_3$. MW, 219·5. B.p. 62–4°/2 mm. D_{20}^{20} 1·3080. n_D^{20} 1·4680.

Natterer, *Monatsh.*, 1883, **4**, 551.
Ladd, Sargent, U.S.P. 2,485,100, (*Chem. Abstracts*, 1950, **44**, 7346).

1 : 2 : 2-Trichlorobutyric Acid

$CH_3{\cdot}CCl_2{\cdot}CHCl{\cdot}COOH$

$C_4H_5O_2Cl_3$ MW, 191·5

Plates from ligroin. M.p. 51·5–52°. Sol. EtOH, Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. H_2O.

Szenic, Taggesell, *Ber.*, 1895, **28**, 2665.

3 : 3 : 3-Trichlorobutyric Acid

$CCl_3{\cdot}CH_2{\cdot}CH_2{\cdot}COOH$

$C_4H_5O_2Cl_3$ MW, 191·5

Needles from H_2O. M.p. 55°.

Me ester: b.p. 78–80°/8 mm.

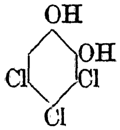
Amide: cryst. from toluene. M.p. 89–90°.

Anhydride: $C_8H_8O_3Cl_6$. MW, 365. B.p. 138–40°/20 mm. D_4^{20} 0·970.

Nitrile: $C_4H_4NCl_3$. MW, 172·5. B.p. 214–16°, 90–5°/12 mm.

Baroni, *Gazz. chim. ital.*, 1933, **63**, 23.
Bruson, Niederhauser, Reiner, Hester, *J. Am. Chem. Soc.*, 1945, **67**, 601.

3 : 4 : 5-Trichlorocatechol



$C_6H_3O_2Cl_3$ MW, 213·5

Two hydrates are known:

(i) + $1H_2O$. Prisms from AcOH. M.p. 115° (104–5°). Sol. EtOH, Et_2O, AcOH. Insol. cold H_2O. $FeCl_3 \longrightarrow$ green col.

(ii) + $\frac{1}{2}H_2O$. Prisms from C_6H_6. M.p. 134–5°.

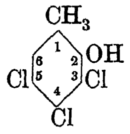
1(or 2)-*Me ether*: trichloroguaiacol. $C_7H_5O_2Cl_3$. MW, 227·5. Needles from pet. ether. M.p. 107–8°. Volatile in steam. *Benzoyl*: cryst. from ligroin. M.p. 128–9°.

2(or 1)-*Me ether*: needles. M.p. 114–15°. Sol. EtOH, Et_2O. Insol. H_2O. Volatile in steam. $FeCl_3 \longrightarrow$ green col.

Di-Me ether: 3 : 4 : 5-trichloroveratrol. $C_8H_7O_2Cl_3$. MW, 241·5. Needles. M.p. 68–9°. Sol. C_6H_6, hot EtOH. Insol. H_2O.

Cousin, *Ann. chim. phys.*, 1903, **29**, 60, 90.
Jackson, Boswell, *Am. Chem. J.*, 1906, **35**, 526.
Peratoner, Ortoleva, *Gazz. chim. ital.*, 1898, **28**, 230.

3 : 4 : 5-Trichloro-*o*-cresol



$C_7H_5OCl_3$ MW, 211·5

Needles from pet. ether. M.p. 77°. Sol. common org. solvents.

Acetyl: needles from MeOH.Aq. M.p. 45°.

Me ether: needles from EtOH. M.p. 51·5°. B.p. 269–71°/752 mm.

Zincke, Preiss, *Ann.*, 1918, **417**, 204.
Kohn, Syreia, *J. Am. Chem. Soc.*, 1948, **70**, 3950.

3 : 5 : 6-Trichloro-*o*-cresol.

Needles from AcOH. M.p. 62°. Sol. common org. solvents except pet. ether.

Benzoyl: needles from EtOH. M.p. 110°.

Zincke, Preiss, *Ann.*, 1918, **417**, 205.

2 : 4 : 6-Trichloro-*m*-cresol.

Needles or plates from H_2O. M.p. 47°. B.p. 265°, 162–3°/28 mm., 142–4°/14 mm. Sol. common org. solvents. Spar. sol. H_2O. Volatile in steam. Complex with cuprammonium sulphate employed as a fungicide.

Me ether: $C_8H_7OCl_3$. MW, 225·5. Needles from AcOH.Aq. M.p. 54–5° (46°).

Et ether: $C_9H_9OCl_3$. MW, 239·5. M.p. 35·5°.

Phenyl ether: $C_{13}H_9OCl_3$. MW, 287·5. M.p. 103°.

Acetyl: cryst. from Et_2O. M.p. 35° (32°). B.p. 273–4°.

Benzoyl: plates. M.p. 53°.

Benzenesulphonyl: plates. M.p. 121°.

p-*Toluenesulphonyl* : plates. M.p. 92–3°.

Huston, Chen, *J. Am. Chem. Soc.*, 1933, **56**, 4218.
Crowther, McCombie, *J. Chem. Soc.*, 1913, **103**, 542.
Raiford, *Am. Chem. J.*, 1911, **46**, 423.
Chulkov, Parini, Barshev, *Org. Chem. Ind. U.S.S.R.*, 1937, **3**, 410, (*Chem. Abstracts*, 1937, **31**, 7047).

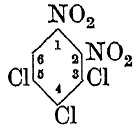
2 : 3 : 5-Trichloro-*p*-cresol.
Needles from AcOH or pet. ether. M.p. 66–7°.
Acetyl : needles from AcOH.Aq. M.p. 37–8°.
Benzoyl : plates from EtOH.Aq. M.p. 89°.

Zincke, *Ann.*, 1903, **328**, 279.

2 : 3 : 6-Trichloro-*p*-cresol.
Cryst. from AcOH. M.p. 85–6°.

Datta, Mitter, *J. Am. Chem. Soc.*, 1919, **41**, 2034.

3 : 4 : 5-Trichloro-*o*-dinitrobenzene



$C_6HO_4N_2Cl_3$ MW, 271·5

Yellow cryst. from EtOH. M.p. 105–6°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 451.

3 : 4 : 6-Trichloro-*o*-dinitrobenzene.
Yellow prisms. M.p. 70–1°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 453.

2 : 4 : 5-Trichloro-*m*-dinitrobenzene.
Pale yellow prisms from EtOH. M.p. 103·5°. B.p. 335°. D^{25} 1·850. Sol. Et_2O, CS_2, C_6H_6, hot EtOH.

Qvist, Salo, *Chem. Zentr.*, 1936, I, 540.
Hüffer, *Rec. trav. chim.*, 1921, **40**, 452.

2 : 4 : 6-Trichloro-*m*-dinitrobenzene.
Prisms from EtOH. M.p. 129·5°. Sol. Me_2CO, $CHCl_3$, CS_2, C_6H_6.

Jackson, Wing, *Am. Chem. J.*, 1887, **9**, 353.
Hüffer, *Rec. trav. chim.*, 1921, **40**, 451.

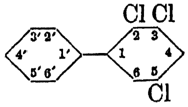
4 : 5 : 6-Trichloro-*m*-dinitrobenzene.
Greenish-yellow needles from EtOH. M.p. 94°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 452.

2 : 3 : 5-Trichloro-*p*-dinitrobenzene.
Yellow needles from EtOH. M.p. 102·5°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 457.

2 : 3 : 5-Trichlorodiphenyl



$C_{12}H_7Cl_3$ MW, 257·5

Needles from EtOH.Aq. M.p. 41°.

Hinkel, Hey, *J. Chem. Soc.*, 1928, 2790.

2 : 5 : 4′-Trichlorodiphenyl.
M.p. 67°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 632.

3 : 4 : 2′-Trichlorodiphenyl.
M.p. 65–6° (54°).

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 632.
Mascarelli, Gatti, Longo, *Gazz. chim. ital.*, 1933, **63**, 654.

3 : 5 : 2′-Trichlorodiphenyl.
Pale yellow needles from EtOH. M.p. 58°.

Hinkel, Hey, *J. Chem. Soc.*, 1928, 2791.

3 : 5 : 4′-Trichlorodiphenyl.
Needles from EtOH. M.p. 88°.

Hinkel, Hey, *J. Chem. Soc.*, 1928, 2791.

1 : 1 : 1-Trichloroethane (*Methylchloroform*)

$CH_3{\cdot}CCl_3$

$C_2H_3Cl_3$ MW, 133·5

F.p. − 32·7°. B.p. 74·0°. D_{25}^{25} 1·31144. n_D^{20} 1·4379.

Städel, *Ann.*, 1879, **195**, 184.
I.G., D.R.P., 523,436, (*Chem. Zentr.*, 1931, I, 3607).
Turkevich, Smyth, *J. Am. Chem. Soc.*, 1940, **62**, 2468.
Nutting, Huscher, U.S.P. 2,209,000, (*Chem. Abstracts*, 1941, **35**, 140).

1 : 1 : 2-Trichloroethane

$CH_2Cl{\cdot}CHCl_2$

$C_2H_3Cl_3$ MW, 133·5

F.p. − 35·5°. B.p. 113·7°, 85·99°/315·5 mm., 49·97°/75·86 mm. D_4^{20} 1·4416.

Prins, *Rec. trav. chim.*, 1926, **45**, 80.
Dreisbach, Shrader, *Ind. Eng. Chem.*, 1949, **41**, 2879.

2 : 2 : 2-Trichloroethyl Alcohol

$CCl_3{\cdot}CH_2OH$

$C_2H_3OCl_3$ MW, 149·5

Plates. M.p. 19°. B.p. 151°, 94–7°/125 mm. Very hygroscopic. Misc. in all proportions with EtOH, Et_2O. Spar. sol. H_2O. Sol. aq. KOH. Reduces warm Fehling's. Fuming HNO_3 ⟶ trichloroacetic acid. PCl_5 ⟶ 2 : 2 : 2-trichloroethyl phosphite. PCl_5 ⟶ 2 : 2 : 2-trichloroethylphosphate.
p-*Nitrobenzoyl* : prisms from EtOH. M.p. 71°.
p-*Aminobenzoyl* : needles from pet. ether. M.p. 87°.
Urethane : needles. M.p. 64–5°.
1-*Naphthylurethane* : cryst. from ligroin. M.p. 120°.
Phosphite : oil. B.p. 263°.

Phosphate: cryst. M.p. 73–4°. Sol. Et_2O. Spar. sol. ligroin.

Chalmers, *Organic Syntheses*, 1935, XV, 80.
Dean, Wolf, *J. Am. Chem. Soc.*, 1936, **58**, 332.
Sroog, Chih, Short, Woodburn, *J. Am. Chem. Soc.*, 1949, **71**, 1710.

Trichloroethylene

$CHCl{:}CCl_2$

C_2HCl_3 MW, 131·5

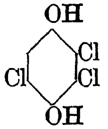
F.p. − 86·4°. M.p. − 73°. B.p. 88–90°, 25°/73 mm. D^{15} 1·4397. n_D^{20} 1·47820. Non-inflammable and employed as a germicide and industrial solvent especially for fats, oils, etc. Resembles chloroform in odour.

Igi, *J. Chem. Ind. Japan*, 1920, **23**, 1217.
Clayton Aniline Co., D.R.P., 222,622, (*Chem. Zentr.*, 1910, II, 121).
Sastry, *Chem. Zentr.*, 1916, II, 306.

Trichloroguaiacol.
See under Trichlorocatechol.

Trichlorohydrin.
See 1 : 2 : 3-Trichloropropane.

Trichlorohydroquinone



$C_6H_3O_2Cl_3$ MW, 213·5

Prisms from H_2O. M.p. 138°. Sol. EtOH, Et_2O. Sol. 160 parts H_2O at 16°. Sublimes in plates.

Di-Et ether: 2 : 3 : 5-trichloro-1 : 4-diethoxy-benzene. $C_{10}H_{11}O_2Cl_3$. MW, 269·5. Needles from EtOH. M.p. 68·5°.

Diacetyl: cryst. from EtOH. M.p. 155–60°.

Conant, Fieser, *J. Am. Chem. Soc.*, 1923, **45**, 2206.
Graebe, *Ann.*, 1868, **146**, 27.
Levy, Schultz, *Ann.*, 1881, **210**, 153.
Oliverio, *Gazz. chim. ital.*, 1948, **78**, 105.

2 : 2 : 2-Trichloro-1-hydroxy-acetylpropylamine.
See Chloralacetamide.

ω-Trichloro-γ-hydroxybutyrophenone.
See Chloralacetophenone.

2 : 2 : 2-Trichloro-1-hydroxyethylacetamide.
See Chloralacetamide.

2 : 2 : 2-Trichloro-1-hydroxyethylformamide.
See Chloralamide.

5 : 5 : 5-Trichloro-4-hydroxy-2-ketopentane.
See Chloralacetone.

3 : 3 : 3-Trichloro-2-hydroxypropyl phenyl Ketone.
See Chloralacetophenone.

2 : 2 : 3-Trichloro-1-hydroxyvaleric Acid

$CH_3{\cdot}CHCl{\cdot}CCl_2{\cdot}CH(OH){\cdot}COOH$

$C_5H_7O_3Cl_3$ MW, 221·5

Tablets. M.p. 140°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6. Prac. insol. H_2O.

Et ester: $C_7H_{11}O_3Cl_3$. MW, 249·5. Prisms. M.p. 40°. B.p. 255° decomp.

Amide: $C_5H_8O_2NCl_3$. MW, 220·5. Cryst. from C_6H_6 or pet. ether. M.p. 119°. Sol. EtOH, Et_2O. Spar. sol. H_2O, C_6H_6.

Nitrile: butylchloral cyanhydrin. $C_5H_6ONCl_3$. MW, 202·5. Plates from aq. HCl. M.p. 101–2°. B.p. 230° decomp. Sol. EtOH, Et_2O. Spar. sol. H_2O, C_6H_6. *Acetyl*: yellow oil. B.p. 240–52° decomp.

Acetyl: needles + $1H_2O$. M.p. 84°. Anhyd. comp. is a syrup.

Pinner, Klein, *Ber.*, 1878, **11**, 1488.
Pinner, Bischoff, *Ann.*, 1875, **179**, 99.

1 : 1 : 1-Trichloroisopropyl Alcohol
(*Isopral*)

$CH_3{\cdot}CH(OH){\cdot}CCl_3$

$C_3H_5OCl_3$ MW, 163·5

M.p. 50–1°. B.p. 161·8°/773 mm. Sol. ord. org. solvents. Spar. sol. H_2O. Resembles camphor in odour.

Bayer, D.R.P., 151,545, (*Chem. Zentr.*, 1904, I, 1586).
Garzarolli-Thurnlackh, *Ann.*, 1881, **210**, 77.
Serantes, *Anales asoc. quím. Argentina*, 1924, **12**, 199 (*Bibl.*).

2 : 2 : 2-Trichlorolactic Acid

$CCl_3{\cdot}CH(OH){\cdot}COOH$

$C_3H_3O_3Cl_3$ MW, 193·5

Prisms. M.p. anhyd. 124°. B.p. 140–70°/45 mm. Sol. H_2O, EtOH, Et_2O, $CHCl_3$. $k = 4{\cdot}65 \times 10^{-3}$ at 25°.

Me ester: $C_4H_5O_3Cl_3$. MW, 207·5. Liq. B.p. 98–100°/12 mm.

Et ester: $C_5H_7O_3Cl_3$. MW, 221·5. Plates. M.p. 66–7°. B.p. 233–7°. Insol. H_2O. Sol. alkalis. *Et ether*: $C_7H_{11}O_3Cl_3$. MW, 249·5. Liq. B.p. 128–30°/12 mm. D_4^{20} 1·34115. *Acetyl*: b.p. 121–121·5°/16 mm. D^{20} 1·367. *Urethane*: needles from Et_2O–pet. ether. M.p. 57·5°.

Propyl ester: $C_6H_9O_3Cl_3$. MW, 235·5. B.p. 248–50°, 115–17°/12 mm. D_4^{20} 1·51628.

Isobutyl ester: $C_7H_{11}O_3Cl_3$. MW, 249·5. B.p. 236–8°, 111–12°/12 mm. D_4^{20} 1·53216.

Amide: $C_3H_4O_2NCl_3$. MW, 192·5. Needles. M.p. 96°. Sol. EtOH, Et_2O. Spar. sol. cold H_2O. O-*Acetyl*: needles. M.p. 94–5°.

Nitrile: chloral cyanhydrin. $C_3H_2ONCl_3$. MW, 174·5. Tablets from CS_2 or H_2O. M.p. 61°. B.p. 215–20° slight decomp. Sol. H_2O, EtOH, Et_2O. *Acetyl*: cryst. M.p. 31°. B.p.

208°. *Urethane*: tablets from EtOH.Aq. M.p. 115–16°.

Anilide: prisms from $CHCl_3$. M.p. 164–5° decomp.

Acetyl: cryst. from C_6H_6. M.p. 65°.

Kölln, *Ann.*, 1918, **416**, 232.
Anschütz, Haslam, *Ann.*, 1889, **253**, 129.
Pinner, *Ber.*, 1884, **17**, 1997.

Trichloromethane.
See Chloroform.

Trichloromethyl bromide.
See Trichlorobromomethane.

Trichloromethyl chloroformate (*Diphosgene, perchloromethyl chloroformate, superpalite*)

$ClCO{\cdot}OCCl_3$

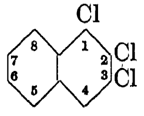
$C_2O_2Cl_4$ MW, 198

Oily liq. B.p. 128°, 49°/50 mm. D^{14} 1·6525. n_D^{22} 1·4566. Decomp. on strong heating to $COCl_2$. Very toxic and asphyxiating. Used during 1st World War as a poison gas under the name of Green Cross.

Grignard, Rivat, Urbain, *Compt. rend.*, 1919, **169**, 1075, 1143.
Kling, Florentin, Lassieur, Schmutz, *Compt. rend.*, 1919, **169**, 1166.

Trichloromethyl sulphochloride.
See Perchloromethyl Mercaptan.

1 : 2 : 3-Trichloronaphthalene



$C_{10}H_5Cl_3$ MW, 231·5

Prisms from Et_2O–EtOH. M.p. 81°.

Faust, Saame, *Ann.*, 1871, **160**, 71.
Armstrong, Wynne, *Chem. News*, 1890, **61**, 272.

1 : 2 : 4-Trichloronaphthalene.
Needles. M.p. 92°.

Cleve, *Ber.*, 1890, **23**, 954.
Armstrong, Wynne, *Chem. News*, 1890, **61**, 273.

1 : 2 : 5-Trichloronaphthalene.
Needles from EtOH. M.p. 78–78·5°. Sol. EtOH, $CHCl_3$, AcOH, C_6H_6.

Armstrong, Wynne, *Chem. News*, 1889, **59**, 188; 1890, **62**, 164.

1 : 2 : 6-Trichloronaphthalene.
Needles from EtOH. M.p. 92·5°. Sol. $CHCl_3$.

Armstrong, Wynne, *Chem News*, 1889, **59**, 189; 1890, **61**, 274.

1 : 2 : 7-Trichloronaphthalene.
Needles from EtOH. M.p. 88°.

Armstrong, Wynne, *Chem. News*, 1889, **59**, 189; 1895, **71**, 254.

1 : 2 : 8-Trichloronaphthalene.
Needles from EtOH. M.p. 83°.

Armstrong, Wynne, *Chem. News*, 1895, **71**, 253.

1 : 3 : 5-Trichloronaphthalene.
Yellow needles from EtOH. M.p. 103° (94°). Sol. common org. solvents.

Friedländer, Karamessinis, Schenk, *Ber.*, 1922, **55**, 49.
Armstrong, Wynne, *Chem. News*, 1890, **61**, 273; 1896, **73**, 55.

1 : 3 : 6-Trichloronaphthalene.
Needles. M.p. 80·5°.

Armstrong, Wynne, *Chem. News*, 1890, **62**, 164; 1895, **71**, 254.

1 : 3 : 7-Trichloronaphthalene.
Needles from EtOH. M.p. 113°. Mod. sol. EtOH.

Armstrong, Wynne, *Chem. News*, 1890, **61**, 93, 275; 1897, **76**, 69.

1 : 3 : 8-Trichloronaphthalene.
Needles from EtOH. M.p. 89·5°.

Armstrong, Wynne, *Chem. News*, 1890, **61**, 94.

1 : 4 : 5-Trichloronaphthalene.
Needles. M.p. 131°. Sol. AcOH, warm EtOH.

Armstrong, Wynne, *Chem. News*, 1890, **61**, 273.
I.G., F.P., 683,792, (*Chem. Zentr.*, 1930, II, 1446).

1 : 4 : 6-Trichloronaphthalene.
Needles from EtOH. M.p. 65°. Spar. sol. boiling EtOH.

Armstrong, Wynne, *Chem. News*, 1890, **61**, 94, 275; 1890, **62**, 162.

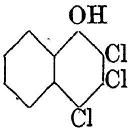
1 : 6 : 7-Trichloronaphthalene.
Needles. M.p. 109·5°.

Armstrong, Wynne, *Chem. News*, 1890, **61**, 275; 1895, **71**, 253.

2 : 3 : 6-Trichloronaphthalene.
Cryst. from MeOH–EtOH. M.p. 145° (90·5–91°).

Franzen, Stäuble, *J. prakt. Chem.*, 1922, **103**, 377.
Armstrong, Wynne, *Chem. News*, 1890, **61**, 275; 1890, **62**, 163.

2 : 3 : 4-Trichloro-1-naphthol



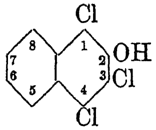
$C_{10}H_5OCl_3$ MW, 247·5

Needles from ligroin. M.p. 168° (159–60°). Sol. Et_2O. Mod. sol. AcOH, warm EtOH.

Acetyl: needles. M.p. 123–4°.

Franzen, Stäuble, *J. prakt. Chem.*, 1922, **103**, 385.
Zincke, *Ber.*, 1888, **21**, 1036.

1 : 3 : 4-Trichloro-2-naphthol



$C_{10}H_5OCl_3$ MW, 247·5

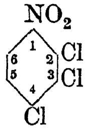
Needles. M.p. 162°.
Acetyl: needles from AcOH. M.p. 133·5–134°.

Zincke, *Ber.*, 1888, **21**, 3390, 3554.

1 : 4 : 5-Trichloro-2-naphthol.
Needles from AcOH. M.p. 157–8°.
Acetyl: m.p. 129°.

Armstrong, Rossiter, *Chem. News*, 1891, **63**, 136.

2 : 3 : 4-Trichloronitrobenzene



$C_6H_2O_2NCl_3$ MW, 226·5

Needles. M.p. 55·5°. Sol. CS_2. Spar. sol. EtOH.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.
Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 69.

2 : 3 : 5-Trichloronitrobenzene.
Yellow needles from 80% EtOH. M.p. 45°.

Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2920.
Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 72.

2 : 3 : 6-Trichloronitrobenzene.
Needles from EtOH. M.p. 88–9°. Sol. EtOH. Spar. sol. ligroin.

Beilstein, Kurbatow, *Ann.*, 1878, **192**, 232.
Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 69.

2 : 4 : 5-Trichloronitrobenzene.
Prisms from EtOH or CS_2. M.p. 57°. B.p. 288°. D^{22} 1·790. Sol. Et_2O, C_6H_6, CS_2. Spar. sol. EtOH.

Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 71.
Cohen, Bennett, *J. Chem. Soc.*, 1905, **87**, 321.

2 : 4 : 6-Trichloronitrobenzene.
Needles from EtOH. M.p. 69°. Spar. sol. EtOH.

Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 74.

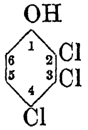
3 : 4 : 5-Trichloronitrobenzene.
Pale yellow cryst. M.p. 72·5°. Spar. volatile in steam.

Holleman, *Rec. trav. chim.*, 1921, **40**, 69.

Trichloronitromethane.
See Chloropicrin.

Trichlorophenetole.
See under Trichlorophenol.

2 : 3 : 4-Trichlorophenol



$C_6H_3OCl_3$ MW, 197·5

Needles from ligroin. M.p. 83·5°. $k = 2{\cdot}22 \times 10^{-8}$ at 25°.
Me ether: 2 : 3 : 4-trichloroanisole. $C_7H_5OCl_3$. MW, 211·5. Prisms from EtOH. M.p. 69·5°.
Benzoyl: needles from EtOH. M.p. 141°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 743.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.
Hodgson, Kershaw, *J. Chem. Soc.*, 1930, 1421.

2 : 3 : 5-Trichlorophenol.
Cryst. M.p. 62°. Very hygroscopic. $k = 4{\cdot}33 \times 10^{-8}$ at 25°.
Me ether: 2 : 3 : 5-trichloroanisole. Needles from EtOH. M.p. 84°.
Benzoyl: needles from ligroin. M.p. 103°.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 114.
Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2921.

2 : 3 : 6-Trichlorophenol.
Needles from pet. ether. M.p. 58°. $k = 7{\cdot}37 \times 10^{-7}$ at 25°.
Benzoyl: cryst. from EtOH. M.p. 90°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 742.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 113.

2 : 4 : 5-Trichlorophenol.
Needles from pet. ether. M.p. 68°. B.p. 244–8°/746 mm. $k = 3{\cdot}76 \times 10^{-8}$ at 25°.
Me ether: 2 : 4 : 5-trichloroanisole. Needles from EtOH. M.p. 77·5°. B.p. 252–5°/742 mm.
Formyl: needles. M.p. 72–3°.
Acetyl: needles from MeOH. M.p. 67–8°.
Propionyl: m.p. 38°. B.p. 123–30°/5 mm. D_{25}^{25} 1·3902. n_D^{20} 1·5418.
Caproyl: b.p. 166°/4 mm. D_{25}^{25} 1·2691. n_D^{20} 1·5250.
Benzoyl: needles from EtOH. M.p. 92–3°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 737.
Kohn, Fink, *Monatsh.*, 1931, **58**, 83.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 114.
Cade, Gump, U.S.P. 2,457,805, (*Chem. Abstracts*, 1949, **43**, 3459).

2 : 4 : 6-Trichlorophenol.

Needles from AcOH. M.p. 69·5°. B.p. 246°. D_4^{75} 1·4901. $k = 3·76 \times 10^{-8}$ at 25°. Sol. EtOH, Et_2O. Volatile in steam.

Me ether: 2 : 4 : 6-trichloroanisole. Needles from EtOH. M.p. 61–2°. B.p. 240°/738 mm. Sublimes.

Et ether: 2 : 4 : 6-trichlorophenetole. $C_8H_7OCl_3$. MW, 225·5. Prisms. M.p. 43–4°. B.p. 246°.

Acetyl: b.p. 261–2°.

Propionyl: b.p. 262·5–264·5°.

Butyryl: b.p. 272–5°.

Benzoyl: needles from EtOH. M.p. 75·5°.

p-*Nitrobenzoyl*: m.p. 105–6°.

p-*Iodophenylurethane*: m.p. 172–3°.

Kohn, Fink, *Monatsh.*, 1931, **58**, 88.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 115.
Datta, Mitter, *J. Am. Chem. Soc.*, 1919, **41**, 2032.
Sah, Young, *Rec. trav. chim.*, 1940, **59**, 357.

3 : 4 : 5-Trichlorophenol.

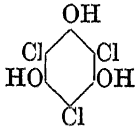
Needles from ligroin. M.p. 101° (91°). B.p. 271–7°/746 mm. $k = 1·77 \times 10^{-8}$ at 25°.

Me ether: 3 : 4 : 5-trichloroanisole. M.p. 63°. B.p. 256–61°.

Benzoyl: needles from EtOH. M.p. 120°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 740.
Tiessens, *Rec. trav. chim.*, 1931, **50**, 113.
Kohn, Kramer, *Monatsh.*, 1928, **49**, 161.

Trichlorophloroglucinol



$C_6H_3O_3Cl_3$ MW, 229·5

Cryst. from EtOH. M.p. 136°. Sol. EtOH. Prac. insol. H_2O, cold C_6H_6.

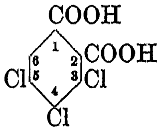
Di-Me ether: $C_8H_7O_3Cl_3$. MW, 257·5. Cryst. from C_6H_6. M.p. 93–5°. *Acetyl*: m.p. 58–9°.

Tri-Me ether: $C_9H_9O_3Cl_3$. MW, 271·5. Needles from EtOH. M.p. 130–1°. Volatile in steam.

Triacetyl: plates from AcOH.Aq. M.p. 167–8°.

Zincke, Kegel, *Ber.*, 1889, **22**, 1476.

3 : 4 : 5-Trichlorophthalic Acid



$C_8H_3O_4Cl_3$ MW, 269·5

Yellow cryst. Heat ⟶ anhydride.

Anhydride: $C_8HO_3Cl_3$. MW, 251·5. M.p. 157°. Sublimes in needles.

Claus, Kautz, *Ber.*, 1885, **18**, 1370.

3 : 4 : 6-Trichlorophthalic Acid.

Cryst. from H_2O. Above 130° ⟶ anhydride.

Anhydride: m.p. 148°. Sublimes in needles. Hot H_2O ⟶ acid.

Imide: $C_8H_2O_2NCl_3$. MW, 250·5. Needles from EtOH. M.p. 236°. Sol. EtOH, Et_2O. Mod. sol. hot H_2O.

Graebe, Rostowzew, *Ber.*, 1901, **34**, 2107.

1 : 1 : 1-Trichloropropane

$CH_3·CH_2·CCl_3$

$C_3H_5Cl_3$ MW, 147·5

B.p. 145–50°. Heat with $Ag_2O + H_2O$ ⟶ propionic acid.

v. Arkel, *Rec. trav. chim.*, 1932, **51**, 1101.
Spring, Lecrenier, *Bull. soc. chim.*, 1887, **48**, 625.

1 : 1 : 2-Trichloropropane (*α-Chloropropylidene chloride*)

$CH_3·CHCl·CHCl_2$

$C_3H_5Cl_3$ MW, 147·5

Oil. B.p. 140° (132°). D^{25} 1·372.

Klebanski, Wolkenstein, *Chem. Zentr.*, 1935, II, 3298.
Herzfelder, *Ber.*, 1893, **26**, 1258.

1 : 1 : 3-Trichloropropane (*β-Chloropropylidene chloride*)

$CH_2Cl·CH_2·CHCl_2$

$C_3H_5Cl_3$ MW, 147·5

B.p. 146–8°. D^{15} 1·362.

Kirrman, Pacaud, Dosque, *Bull. soc. chim.*, 1934, **1**, 864.
Romburgh, *Bull. soc. chim.*, 1882, **37**, 100.

1 : 2 : 2-Trichloropropane (*Chloroisopropylidene chloride*)

$CH_3·CCl_2·CH_2Cl$

$C_3H_5Cl_3$ MW, 147·5

B.p. 123°. D^{25} 1·318.

Herzfelder, *Ber.*, 1893, **26**, 1259.

1 : 2 : 3-Trichloropropane (*Trichlorohydrin, glycerol trichlorohydrin*)

$CH_2Cl·CHCl·CH_2Cl$

$C_3H_5Cl_3$ MW, 147·5

B.p. 158°. D^{15} 1·417.

Carré, Mauclère, *Compt. rend.*, 1931, **192**, 1568.
Fittig, Pfeffer, *Ann.*, 1865, **135**, 359.
Kharasch, Brown, *J. Am. Chem. Soc.*, 1939, **61**, 3432.

1 : 1 : 2-Trichloropropionic Acid

$CH_2Cl·CCl_2·COOH$

$C_3H_3O_2Cl_3$ MW, 177·5

Prisms from CS_2. M.p. 65–6°. B.p. 140°/40 mm. Sol. H_2O, EtOH, C_6H_6.

Me ester: b.p. 102–4°/55 mm. D_{15}^{28} 1·446.

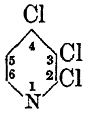
Et ester : $C_5H_7O_2Cl_3$. MW, 205·5. B.p. 121°/55 mm. D^{25} 1·36. n^{25} 1·458.

Isobutyl ester : b.p. 121–3°/32 mm. D^{28}_{15} 1·233.

Cyclohexyl ester : b.p. 136–49°/19 mm.

Berlande, *Bull. soc. chim.*, 1925, **37**, 1385.
Plump, U.S.P. 2,370,577, (*Chem. Abstracts*, 1945, **39**, 4085).
Lichtz, U.S.P. 2,400,466, (*Chem. Abstracts*, 1946, **40**, 4743); U.S.P. 2,405,894, (*Chem. Abstracts*, 1946, **40**, 7232).

2 : 3 : 4-Trichloropyridine



$C_5H_2NCl_3$ MW, 182·5

Needles. M.p. 45–7°. Sublimes in vacuo.

Graf, *J. prakt. Chem.*, 1933, **138**, 235.

2 : 3 : 5-Trichloropyridine.

Needles from 50% EtOH. M.p. 50°. Sol. Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Mod. sol. EtOH, pet. ether. Insol. H_2O, dil. acids.

Räth, *Ann.*, 1931, **486**, 78.
Fischer, Chur, *J. prakt. Chem.*, 1916, **93**, 371.

2 : 3 : 6-Trichloropyridine.

Cryst. from EtOH.Aq. M.p. 66–7°.

Crouch, Lochte, *J. Am. Chem. Soc.*, 1943, **65**, 270.

2 : 4 : 6-Trichloropyridine.

M.p. 33°.

Graf, *J. prakt. Chem.*, 1932, **133**, 44.

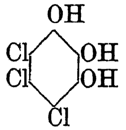
3 : 4 : 5-Trichloropyridine.

Needles from EtOH.Aq. M.p. 76–7°. Sol. Et_2O, $CHCl_3$, C_6H_6, pet. ether. Mod. sol. EtOH. Spar. sol. H_2O. Sol. conc. min. acids.

B_2,$HgCl_2$: needles. M.p. 168–70°.

Dohrn, Diedrich, *Ann.*, 1932, **494**, 298.
Sell, *J. Chem. Soc.*, 1905, **87**, 800.

Trichloropyrogallol



$C_6H_3O_3Cl_3$ MW, 229·5

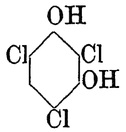
Needles + $3H_2O$. M.p. anhyd. 185° (177°). Sol. EtOH, Et_2O, hot H_2O. Spar. $CHCl_3$, CCl_4, CS_2, C_6H_6.

Tri-Me ether : $C_9H_9O_3Cl_3$. MW, 271·5. Cryst. from EtOH. M.p. 54°. Spar. volatile in steam.

Triacetyl : needles from AcOH.Aq. M.p. 122°.

Hantzsch, Schniter, *Ber.*, 1887, **20**, 2036.
Webster, *J. Chem. Soc.*, 1884, **45**, 205.

2 : 4 : 6-Trichlororesorcinol



$C_6H_3O_2Cl_3$ MW, 213·5

Cryst. from H_2O. M.p. 83°. Sol. EtOH, Et_2O. Spar. sol. H_2O.

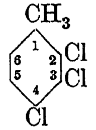
Diacetyl : prisms from EtOH. M.p. 116°.

Likhosherstov, *Chem. Abstracts*, 1934, **28**, 1675.
Zincke, Rabinowitsch, *Ber.*, 1890, **23**, 3767.
Coveti, U.S.P. 2,485,562, (*Chem. Abstracts*, 1950, **44**, 3022).

ω-Trichlorotoluene.

See Benzotrichloride.

2 : 3 : 4-Trichlorotoluene



$C_7H_5Cl_3$ MW, 195·5

Needles from EtOH. M.p. 41°. B.p. 231–2°/716 mm. Sol. common org. solvents. Dil. HNO_3 ⟶ 2 : 3 : 4-trichlorobenzoic acid.

Seelig, *Ann.*, 1887, **237**, 132, 156.

2 : 3 : 5-Trichlorotoluene.

Needles from EtOH. M.p. 45–6°. B.p. 229–31°/757 mm. Dil. HNO_3 ⟶ 2 : 3 : 5-trichlorobenzoic acid.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1343.

2 : 3 : 6-Trichlorotoluene.

Needles from EtOH. M.p. 45–6°. Dil. HNO_3 ⟶ 2 : 3 : 6-trichlorobenzoic acid.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1343.

2 : 4 : 5-Trichlorotoluene.

Needles from EtOH. M.p. 82°. B.p. 229–30°/716 mm., 152°/25 mm. Dil. HNO_3 ⟶ 2 : 4 : 5-trichlorobenzoic acid. Sublimes in needles.

Fichter, Glanztein, *Ber.*, 1916, **49**, 2481.
Feldman, Kopeliowitsch, *Chem. Zentr.*, 1936, I, 2550.
Qvist, Holmberg, *Chem. Zentr.*, 1932, II, 2816.
B.D.C., B.P. 169,025, (*Chem. Zentr.*, 1922, IV, 376).

2 : 4 : 6-Trichlorotoluene.

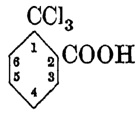
Needles from EtOH. M.p. 38° (33–4°). Volatile in steam. Dil. HNO_3 ⟶ 2 : 4 : 6-trichlorobenzoic acid.

Bureš, Trpišovska, *Chem. Zentr.*, 1936, I, 1209.
Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1335.

3 : 4 : 5-Trichlorotoluene.
M.p. 44·5–45·5°. B.p. 245·5–247°. Volatile in steam. Dil. $HNO_3 \longrightarrow$ 3 : 4 : 5-trichlorobenzoic acid.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1337.

ω-Trichloro-o-toluic Acid (*Benzotrichloride-o-carboxylic acid*)



$C_8H_5O_2Cl_3$ MW, 239·5

Needles from C_6H_6. M.p. 141–4°.
Me ester: $C_9H_7O_2Cl_3$. MW, 253·5. B.p. 125°/1 mm.
Et ester: $C_{10}H_9O_2Cl_3$. MW, 267·5. Mobile liq. Decomp. on dist.
Chloride: $C_8H_4OCl_4$. MW, 258. Cryst. from pet. ether. M.p. 87°. B.p. 145–55°/20 mm.
Nitrile: o-cyanobenzotrichloride. $C_8H_4NCl_3$. MW, 220·5. Cryst. from EtOH. M.p. 94–5°. B.p. 280°. Conc. HCl in sealed tube $\longrightarrow$ phthalic acid.
Anilide: cryst. from C_6H_6. M.p. 165–70°.

Davies, Perkin, *J. Chem. Soc.*, 1922, **121**, 2213.
Ott, *Ber.*, 1922, **55**, 2123.

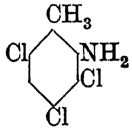
ω-Trichloro-m-toluic Acid (*Benzotrichloride-m-carboxylic acid*).
Plates. M.p. 142°. Sol. $CHCl_3$, C_6H_6. Spar. sol. formic acid.
Me ester: needles from Me_2CO–MeOH. M.p. 55°.
Chloride: oil. B.p. 287°/754 mm.

Davies, Perkin, *J. Chem. Soc.*, 1922, **121**, 2213.

ω-Trichloro-p-toluic Acid (*Benzotrichloride-p-carboxylic acid*).
Plates. M.p. 196–7°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. formic acid.
Me ester: plates. M.p. 55°.
Et ester: plates. M.p. 57°.
Chloride: oil. B.p. 296°/756 mm.
Amide: $C_8H_6ONCl_3$. MW, 238·5. Needles from C_6H_6. M.p. 180°.

Davies, Perkin, *J. Chem. Soc.*, 1922, **121**, 2214.
Böeseken, Gelissen, *Rec. trav. chim.*, 1924, **43**, 869.

3 : 4 : 6-Trichloro-o-toluidine

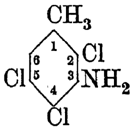


$C_7H_6NCl_3$ MW, 210·5

Needles from EtOH. M.p. 89°. Volatile in steam.
N-*Acetyl*: 3 : 4 : 6-trichloroaceto-o-toluidide. Needles from EtOH.Aq. M.p. 199°.
N-*Benzoyl*: plates from EtOH. M.p. 230°.

Levy, Stephen, *J. Chem. Soc.*, 1931, 78.

2 : 4 : 5-Trichloro-m-toluidine



$C_7H_6NCl_3$ MW, 210·5

Needles from EtOH. M.p. 94–5°.
N-*Acetyl*: 2 : 4 : 5-trichloroacet-m-toluidide. Needles from EtOH. M.p. 190–1°.
N-*Benzoyl*: needles. M.p. 213°. Spar. sol. hot EtOH.

Seelig, *Ann.*, 1887, **237**, 141.
Schultz, *Ann.*, 1877, **187**, 278.

2 : 4 : 6-Trichloro-m-toluidine.
Needles from EtOH. M.p. 85° (77–8°).
N-*Acetyl*: 2 : 4 : 6-trichloroacet-m-toluidide. Needles from EtOH. M.p. 192° (181°).
N-*Diacetyl*: m.p. 81–2°.
N-*Benzoyl*: leaflets. M.p. 218°.

Bureš, Trpišovska, *Chem. Zentr.*, 1936, I, 1209.
Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1335.

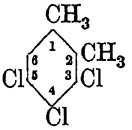
2 : 5 : 6-Trichloro-m-toluidine.
Cryst. from MeOH.Aq. M.p. 66–7°.

Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1281.

2 : 2′ : 2″-Trichlorotriethylamine.
See under 2-Chloroethylamine.

3 : 4 : 5-Trichloroveratrol.
See under 3 : 4 : 5-Trichlorocatechol.

3 : 4 : 5-Trichloro-o-xylene



$C_8H_7Cl_3$ MW, 209·5

Needles from EtOH. M.p. 96°. B.p. 261°. Sol. Et_2O, $CHCl_3$, C_6H_6, pet. ether, hot EtOH. Volatile in steam.

Hinkel, *J. Chem. Soc.*, 1920, **117**, 1301.
Cf. Claus, Kautz, *Ber.*, 1885, **18**, 1369.

3 : 5 : 6-Trichloro-o-xylene.
Cryst. from EtOH. M.p. 47·5°. B.p. 230–40°. Sol. Et_2O, $CHCl_3$, AcOEt, C_6H_6, pet. ether. Mod. sol. MeOH, EtOH.

Hinkel, *J. Chem. Soc.*, 1920, **117**, 1300.

2 : 4 : 5-Trichloro-*m*-xylene.
M.p. 95–6°. B.p. 225–60°.

I.G., F.P., 650,732, (*Chem. Abstracts*, 1929, **23**, 3233).
General Aniline Works Inc., U.S.P., 1,796,108, (*Chem. Zentr.*, 1931, I, 3610).
Cf. Bureš, Borgmann, *Chem. Zentr.*, 1928, I, 1171.

2 : 3 : 5-Trichloro-*p*-xylene.
Needles. M.p. 96°. B.p. 254–5°. Sol. EtOH, Et_2O, C_6H_6, pet. ether. Insol. H_2O.

Bureš, Rubeš, *Chem. Zentr.*, 1929, I, 507.
Cass, U.S.P. 2,412,389, (*Chem. Abstracts*, 1947, **41**, 2079).

Trichothecin

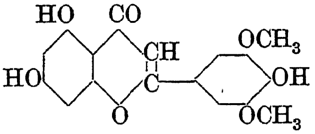
$C_{19}H_{24}O_5$ MW, 332

Antifungal compound from *Trichothecium roseum*, Link. Needles from light petroleum. M.p. 118°. Sol. $CHCl_3$, C_6H_6, EtOH, Me_2CO. Spar. sol. light petroleum, H_2O. $[\alpha]_D^{18}$ + 44°. Alk. hyd. ⟶ isocrotonic acid + a ketonic alcohol, trichothecolone.

2 : 4-*Dinitrophenylhydrazone*: orange needles from EtOH. M.p. 200°.

Freeman, Morrison, *Biochem. J.*, 1949, **44**, 1.
Freeman, Gill, *Nature*, 1950, **166**, 698.

Tricin (*Tricetin 3′ : 5′-dimethyl ether*, 5 : 7 : 4′-*trihydroxy*-3′ : 5′-*dimethoxyflavone*)



$C_{17}H_{14}O_7$ MW, 330

Colouring matter of leaves of Khapli wheat. Yellow needles from AcOH.Aq. M.p. 291–2°. Conc. H_2SO_4 ⟶ yellow sol. Alc. $FeCl_3$ ⟶ reddish-brown col.

4′-Benzyl ether: $C_{24}H_{20}O_7$. MW, 420. Pale orange prismatic needles from Me_2CO.Aq. M.p. 234°. Conc. H_2SO_4 ⟶ pale yellow col. Alc. $FeCl_3$ ⟶ greenish-brown col.

Diacetyl deriv.: pale yellow needles. M.p. 211–13°.

Triacetyl: needles from EtOH–AcOH. M.p. 251–4°.

Tri-Me ether: 5 : 7 : 3′ : 4′ : 5′-pentamethoxyflavone. $C_{20}H_{20}O_7$. MW, 372. Needles from MeOH. M.p. 192–3°.

Anderson, *Chem. Zentr.*, 1932, II, 3899; 1933, II, 2012.
Gulati, Venkataraman, *J. Chem. Soc.*, 1933, 1644.

Tricosane

$CH_3{\cdot}[CH_2]_{21}{\cdot}CH_3$

$C_{23}H_{48}$ MW, 324

Present in Pennsylvania petroleum. Leaflets from EtOH. M.p. 47·3°. B.p. 234°/15 mm, 199·5°/3 mm. D^{90} 0·7431. n_D^{90} 1·4190.

Read, Andrews, *J. Soc. Chem. Ind.*, 1920, **39**, 290T.
Mazee, *Rec. trav. chim.*, 1948, **67**, 197.

Tricosanic Acid

$CH_3{\cdot}[CH_2]_{20}{\cdot}CH_2{\cdot}COOH$

$C_{23}H_{46}O_2$ MW, 354

Cryst. from C_6H_6. M.p. 79·1°.

Me ester: $C_{24}H_{48}O_2$. MW, 368. Cryst. from Me_2CO. M.p. 54·0°.

Et ester: $C_{25}H_{50}O_2$. MW, 382. Cryst. from Me_2CO. M.p. 52–3°. B.p. 198–9°/0·27 mm.

Nitrile: $C_{23}H_{45}N$. MW, 335. Cryst. from Me_2CO. M.p. 53·5–54·5°.

Ashton, Robinson, Smith, *J. Chem. Soc.*, 1936, 285.
Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.
Francis, Piper, *J. Am. Chem. Soc.*, 1939, **61**, 577

Tricosanol-1.
See Tricosyl Alcohol.

Tricosanol-12 (*Diundecylcarbinol*)

$CH_3{\cdot}[CH_2]_{10}{\cdot}CH(OH){\cdot}[CH_2]_{10}{\cdot}CH_3$

$C_{23}H_{48}O$ MW, 340

Cryst. M.p. 75·5°. Sol. AcOH, pet. ether.

Grün, Ulbrich, Krezil, *Z. angew. Chem.*, 1926, **39**, 424.

Tricosanone-12.
See Laurone.

Tricosyl Alcohol (*Tricosanol*-1)

$CH_3{\cdot}[CH_2]_{21}{\cdot}CH_2OH$

$C_{23}H_{48}O$ MW, 340

Cryst. from Me_2CO. M.p. 73·5–74·5°. B.p. 191–3°/0·7 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 915.

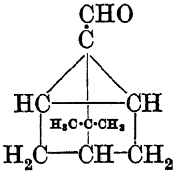
Tricresyl phosphate.
See Tritolyl phosphate.

Tricyanomethane.
See Cyanoform.

1 : 2 : 3-Tricyanopropane.
See under Tricarballylic Acid.

Tricyanotrimethylamine.
See under Triglycolamidic Acid.

Tricyclal

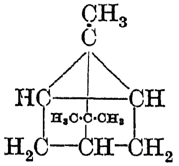


$C_{10}H_{14}O$ MW, 150

M.p. 85–90°. B.p. 113–15°/31 mm.
Semicarbazone: needles from EtOH.Aq. M.p. 219–20° decomp.
Azine: prisms from EtOH. M.p. 171–2°.

Lipp, *Ber.*, 1920, **53**, 778.

Tricyclene (1 : 2 : 2-*Trimethyl*-3 : 6-*methylenebicyclo*-[0, 1, 3]-*hexane, cyclene*)

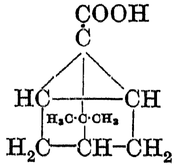


$C_{10}H_{16}$ MW, 136

Cryst. from EtOH. M.p. 64–5° (67–8°). B.p. 152–3°. D^{80} 0·8268. n_{α}^{80} 1·4296.

Nametkin, Zabrodin, *Ann.*, 1925, **441**, 185.
Lipp, *Ber.*, 1920, **53**, 779.
Komppa, *Ber.*, 1929, **62**, 1369.
Chem. Fabrik a. Actien, D.R.P., 353,933, (*Chem. Zentr.*, 1922, IV, 499).

Tricyclenic Acid (*Dehydrocamphenilic acid*)

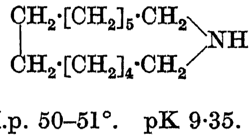


$C_{10}H_{14}O_2$ MW, 166

Plates from EtOH or C_6H_6. M.p. 150–1°. B.p. 262–4°, 145°/12 mm. Sol. EtOH, C_6H_6, pet. ether. Insol. H_2O.
Me ester: $C_{11}H_{16}O_2$. MW, 180. M.p. 45·5° (38°). B.p. 99°/14 mm. $D_4^{42·6}$ 1·0255. $n_D^{42·6}$ 1·46953.
Et ester: $C_{12}H_{18}O_2$. MW, 194. B.p. 100–1°/10 mm. D^{20} 1·0143. n_D^{20} 1·47299.
Chloride: $C_{10}H_{13}OCl$. MW, 184·5. M.p. 37·5–38·5°. B.p. 116–17°/15 mm.
Amide: $C_{10}H_{15}ON$. MW, 165. Leaflets from C_6H_6. M.p. 117–18°.
Nitrile: $C_{10}H_{13}N$. MW, 147. M.p. 65–70°. B.p. 100–2°/12 mm.

Lipp, *Ber.*, 1920, **53**, 774.
Komppa, *Ber.*, 1929, **62**, 1366.
Lipp, Bräucker, *Ber.*, 1939, **72**, 2079.

Tridecamethyleneimine



$C_{13}H_{27}N$ MW, 197

Cryst. M.p. 50–51°. pK 9·35.

Ruzicka, Kobelt, Hafliger, Prelog, *Helv. Chim. Acta*, 1949, **32**, 544.

Tridecanal.
See Tridecyl Aldehyde.

Tridecane

$CH_3·[CH_2]_{11}·CH_3$

$C_{13}H_{28}$ MW, 184

M.p. — 5·5°. B.p. 234°, 130°/30 mm., 114°/15 mm., 92·5°/4·5 mm. D_4^{20} 0·7565, D_4^{70} 0·7209. n_D^{20} 1·42326.

Krafft, *Ber.*, 1882, **15**, 1699.
Schmidt, Schoeller, Eberlein, *Ber.*, 1941, **74**, 1313.
Vogel, *J. Chem. Soc.*, 1946, 133.

Tridecanedioic Acid.
See Brassylic Acid.

Tridecanol-1.
See Tridecyl Alcohol.

Tridecanol-2.
See Methylundecylcarbinol.

Tridecanol-3.
See Ethyl-*n*-decylcarbinol.

Tridecanol-7.
See Di-*n*-hexylcarbinol.

Tridecanone-2.
See Methyl undecyl Ketone.

Tridecanone-3.
See Ethyl *n*-decyl Ketone.

Tridecanone-7.
See Di-*n*-hexyl Ketone.

1-Tridecene (1-*Tridecylene*)

$CH_3·[CH_2]_{10}·CH{:}CH_2$

$C_{13}H_{26}$ MW, 182

M.p. — 22·2°. B.p. 102°/10 mm. D_4^0 0·7856, D_4^{20} 0·7658. n_D^{20} 1·4340, n_D^{25} 1·4328.

Kozacik, Reid, *J. Am. Chem. Soc.*, 1938, **60**, 2436.
Schmidt, Schoeller, Eberlein, *Ber.*, 1941, **74**, 1313.

Tridecene-1-carboxylic Acid.
See Tetradecylenic Acid.

2-Tridecylacrylic Acid.
See Gaidic Acid.

Tridecyl Alcohol (*Tridecanol*-1)

$CH_3·[CH_2]_{11}·CH_2OH$

$C_{13}H_{28}O$ MW, 200

M.p. 30·5°. B.p. 155–6°/15 mm., 117°/0·5 mm.
Propionyl: tridecyl propionate. M.p. — 0·4°. B.p. 195°/30 mm. D_4^{25} 0·8574. n_D^{20} 1·4363.
p-*Nitrobenzoyl*: m.p. 37·4°.
4′-*Iodoxenylurethane*: m.p. 144–144·5°.

Levene, West, Scheer, *J. Biol. Chem.*, 1915, **20**, 528.
Armstrong, Copenhaver, *J. Am. Chem. Soc.*, 1943, **65**, 2252.

Tridecyl Aldehyde (*Tridecanal*)

$CH_3·[CH_2]_{11}·CHO$

$C_{13}H_{26}O$ MW, 198

M.p. 14°. B.p. 126–8°/10 mm. D_4^{18} 0·8356. n_D^{18} 1·4384.

Oxime: needles from EtOH.Aq. M.p. 80·5°.
Semicarbazone: plates from EtOH. M.p. 113–14°.

Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1903.
Stoll, *Helv. Chim. Acta*, 1947, **30**, 122.

Tridecylamine (1-*Aminotridecane*)

$CH_3 \cdot [CH_2]_{11} \cdot CH_2NH_2$

$C_{13}H_{29}N$ MW, 199

M.p. 27°. B.p. 273·8°, 120·8°/4 mm. Sol. EtOH, Et_2O.
Benzoyl: m.p. 70·6°.

Lutz, *Ber.*, 1886, **19**, 1436.
Blau, *Monatsh.*, 1905, **26**, 101.
Ralston *et al.*, *Ind. Eng. Chem.*, 1940, **32**, 1093.
Suida, Drahowzal, *Ber.*, 1942, **75**, 991.

Tridecyl bromide

$CH_3 \cdot [CH_2]_{11} \cdot CH_2Br$

$C_{13}H_{27}Br$ MW, 263

M.p. 6°. B.p. 148–9°/9·5 mm. D_4^{20} 1·0262. n_D^{20} 1·4601.

Suida, Drahowzal, *Ber.*, 1942, **75**, 991.

Tridecyl chloride

$CH_3 \cdot [CH_2]_{11} \cdot CH_2Cl$

$C_{13}H_{27}Cl$ MW, 218·5

B.p. 135·7–136°/9 mm. D_4^{20} 0·8668. n_D^{20} 1·4460.

Suida, Drahowzal, *Ber.*, 1942, **75**, 991.

Tridecylene.
See Tridecene.

1-Tridecylenic Acid (2-n-*Decylacrylic acid*, 1-*dodecylene*-1-*carboxylic acid*)

$CH_3 \cdot [CH_2]_9 \cdot CH{:}CH \cdot COOH$

$C_{13}H_{24}O_2$ MW, 212

B.p. 167–71°/2 mm. D^{30} 0·8995. n_D^{20} 1·46121.
Chloride: $C_{13}H_{23}OCl$. MW, 230·5. B.p. 131°/3 mm. D^{20} 0·9380.
Amide: $C_{13}H_{25}ON$. MW, 211. M.p. 116°.

Zaar, *Chem. Abstracts*, 1930, **24**, 2108.

10-Tridecylenic Acid (*Isotridecylenic acid*, 2-*dodecylene*-12-*carboxylic acid*)

$CH_3 \cdot CH{:}CH \cdot [CH_2]_9 \cdot COOH$

$C_{13}H_{24}O_2$ MW, 212

Plates from pet. ether. M.p. 28–9°. B.p. 183–5°/13·5 mm., 161–2°/4 mm.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 122.

11-Tridecylenic Acid (1-*Dodecylene*-12-*carboxylic acid*)

$CH_2{:}CH \cdot [CH_2]_{10} \cdot COOH$

$C_{13}H_{24}O_2$ MW, 212

Plates from EtOH.Aq. M.p. 38–9°. B.p. 192°/20 mm., 185°/15 mm., 162°/3 mm. Sol. EtOH, Et_2O, C_6H_6, pet. ether.
Me ester: $C_{14}H_{26}O_2$. MW, 226. B.p. 143°/8 mm., 133°/3 mm. D_{20}^{20} 0·8819. n_D^{20} 1·4438.
Et ester: $C_{15}H_{28}O_2$. MW, 240. B.p. 150°/8 mm. D^{15} 0·880.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 118.
Tomecko, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 529.

Tridecylic Acid (*Tridecoic acid*, *dodecane*-1-*carboxylic acid*)

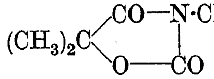
$CH_3 \cdot [CH_2]_{11} \cdot COOH$

$C_{13}H_{26}O_2$ MW, 214

Cryst. from Me_2CO. M.p. 44·5–45·5°. B.p. 199–200°/24 mm.
Zn salt: needles from isoamyl alcohol. M.p. 128°.
Et ester: $C_{15}H_{30}O_2$. MW, 242. B.p. 197–8°/60 mm., 178–80°/20 mm., 163–5°/5 mm.
Propyl ester: $C_{16}H_{32}O_2$. MW, 256. B.p. 194°/30 mm. D_4^{25} 0·8555. n_D^{20} 1·4357.
Amide: $C_{13}H_{27}ON$. MW, 213. M.p. 100°.
Nitrile: $C_{13}H_{25}N$. MW, 195. B.p. 275°.

Ruhoff, *Organic Syntheses*, 1936, XVI, 35.
Levene, West, Allen, Scheer, *J. Biol. Chem.*, 1915, **23**, 73.

Tridione (3 : 5 : 5-*Trimethyloxazolidine*-2 : 4-*dione*)



$C_6H_9O_3N$ MW, 143

Cryst. from 50% MeOH.Aq. M.p. 46°. B.p. 78–80°/5 mm. Very sol. most org. solvents except pet. ether. Part. sol. H_2O. Analgesic.

Spielmann, *J. Am. Chem. Soc.*, 1944, **66**, 1244.

Triethanolamine.
See 2 : 2′ : 2″-Trihydroxytriethylamine.

ω : 2 : 4-Triethoxyacetophenone.
See under Fisetol.

3 : 4 : 5-Triethoxybenzoic Acid.
See under Gallic Acid.

Triethylamine

$(CH_3 \cdot CH_2)_3N$

$C_6H_{15}N$ MW, 101

M.p. — 114·75°. B.p. 89·4°. D_4^{20} 0·7275, $D_4^{60\cdot5}$ 0·6912. n_D^{20} 1·40101. Readily oxidised by $KMnO_4$.
B,HCl: cryst. from EtOH. M.p. 253–4°.
B,HBr: cryst. from $CHCl_3$ or EtOH. M.p. 248°.
B,HI: m.p. 181°. Sol. $CHCl_3$, EtOH.
B,HNO_3: m.p. 99–100°.
B,HBr,$HgBr_2$: m.p. 124–5°.
B_2,3CHI_3: yellow cryst. from EtOH. M.p. 81–3°.
B,H_2S: needles. M.p. 55–7° (closed tube).

Acetate: b.p. 162° decomp.
Picrate: needles from EtOH. M.p. 172–3°.

Skita, Keil, *Monatsh.*, 1929, **53** and **54**, 757.
I.G., B.P. 283,163, (*Chem. Zentr.*, 1929, I, 1509); F.P., 685,345, (*Chem. Zentr.*, 1931, I, 1824).
Hofmann, *Ann.*, 1850, **73**, 91.
Rakshit, *J. Am. Chem. Soc.*, 1913, **35**, 1782.
Vogel, *J. Chem. Soc.*, 1948, 1825.

Triethylarsine (*Arsenic triethyl*)

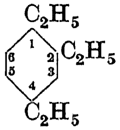
$(C_2H_5)_3As$

$C_6H_{15}As$ MW, 162

B.p. 140°/736 mm. D_4^{20} 1·150. n_D^{20} 1·467. Sol. EtOH, Et_2O. Insol. H_2O. Fumes in air and inflames when heated.

Cahours, *Ann.*, 1862, **122**, 202.
Hofmann, *Ann.*, 1857, **103**, 357.

1 : 2 : 4-Triethylbenzene



$C_{12}H_{18}$ MW, 162

B.p. 217–18°/755 mm. 99°/15 mm. D_4^{17} 0·8819. n_D^{17} 1·4983.

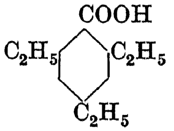
Klages, *J. prakt. Chem.*, 1902, **65**, 398.

1 : 3 : 5-Triethylbenzene.

F.p. − 66·48°. B.p. 215·92°. D_4^{20} 0·8631. n_D^{20} 1·4969. $CrO_3 \longrightarrow$ trimesic acid.

Smith, Guss, *J. Am. Chem. Soc.*, 1940, **62**, 2625.
Huntress, Autenrieth, *J. Am. Chem. Soc.*, 1941, **63**, 3446.

2 : 4 : 6-Triethylbenzoic Acid



$C_{13}H_{18}O_2$ MW, 206

Plates from ligroin. M.p. 113°.
Me ester: $C_{14}H_{20}O_2$. MW, 220. B.p. 93·2–93·8°/0·5–1 mm. n_D^{20} 1·5016.
Butyl ester: $C_{17}H_{26}O_2$. MW, 262. B.p. 136–40°/3 mm.
Chloride: $C_{13}H_{17}OCl$. MW, 224·5. B.p. 112–13°/4 mm. D_4^{20} 1·0424. n_D^{20} 1·5182.
Amide: $C_{13}H_{19}ON$. MW, 205. Needles from ligroin. M.p. 155–6°.

Gattermann, Fritz, Beck, *Ber.*, 1899, **32**, 1123.
Fuson, Corse, *J. Am. Chem. Soc.*, 1938, **60**, 2063.
Newman, *J. Am. Chem. Soc.*, 1941, **63**, 2431.
Newman, Fones, *J. Am. Chem. Soc.*, 1947, **69**, 1046.

Triethyl borate (*Ethyl borate*)

$B(O{\cdot}CH_2{\cdot}CH_3)_3$

$C_6H_{15}O_3B$ MW, 146

Liq. B.p. 118·6°. D^{26} 0·864. n_D 1·3808. Rapidly hyd. by H_2O.

Khotinskii, Pupko, *Chem. Zentr.*, 1929, II, 2763.

Triethylcarbinol (*3-Ethylpentanol-3*)

$(C_2H_5)_3C{\cdot}OH$

$C_7H_{16}O$ MW, 116

Liq. with penetrating camphor-like odour. B.p. 140–2°. $D_4^{22\cdot4}$ 0·8407. $n_D^{22\cdot3}$ 1·4266.
Acetyl: b.p. 153–60°.
Butyryl: b.p. 83–6°/13 mm.
Phenylacetyl: b.p. 142–6°/13 mm.
Allophanate: m.p. 182–3°.

Moyer, Marvel, *Organic Syntheses*, 1931, XI, 98.
Böeseken, Wildschut, *Rec. trav. chim.*, 1932, **51**, 168.
Edgar, Calingaert, Marker, *J. Am. Chem. Soc.*, 1929, **51**, 1486.
Spassow, *Ber.*, 1937, **70**, 1926.

Triethyl citrate

$CH_2{\cdot}CO{\cdot}OC_2H_5$
$C(OH){\cdot}CO{\cdot}OC_2H_5$
$CH_2{\cdot}CO{\cdot}OC_2H_5$

$C_{12}H_{20}O_7$ MW, 276

Oil. B.p. 294°, 230°/100 mm., 212°/30–5 mm. D_4^{20} 1·1369. n_D^{20} 1·44554.
Et ether: $C_{14}H_{24}O_7$. MW, 304. Oil. B.p. 237–8°/150 mm. D_4^{20} 1·1022. n_D^{20} 1·4484.
Acetyl: 2-acetoxytricarballylic triethyl ester. B.p. 197°/15 mm. Sol. EtOH, Et_2O.

Conen, *Ber.*, 1879, **12**, 1653.

Triethylene Glycol (*Ethylene glycol di-β-hydroxyethyl ether, di-β-hydroxyethoxyethane*)

$HO{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2OH$

$C_6H_{14}O_4$ MW, 150

Liq. B.p. 278·31°, 165°/14 mm., 134°/2 mm. D_4^{15} 1·1274. n_D^{15} 1·4578. Misc. with H_2O, EtOH. Spar. sol. Et_2O.

Matignon, Moureau, Dodé, *Bull. soc. chim.*, 1934, **1**, 1311.
Gallaugher, Hibbert, *J. Am. Chem. Soc.*, 1937, **59**, 2514.

Triethylenetetramine (*N:N'-Di-[2-aminoethyl]-ethylenediamine, 1:2-di-[aminoethylamino]-ethane*)

$NH_2CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2NH_2$

$C_6H_{18}N_4$ MW, 146

B.p. 174°/31 mm., 157°/20 mm.
B,4HCl: needles from EtOH–HCl. M.p. 266–70°.
Hydrogen oxalyl: needles from H_2O. Decomp. at 243° corr.

Tetrabenzoyl : micro-cryst. from $CHCl_3$–EtOH. M.p. 238°.
Tetrapicrate : prisms from H_2O. Decomp. at 240° corr.

Peacock, *J. Chem. Soc.*, 1936, 1518.
van Alphen, *Rec. trav. chim.*, 1936, **55**, 412.
Fargher, *J. Chem. Soc.*, 1920, **117**, 1351.

Triethylmethane.
See 3-Ethylpentane.

Triethyl phosphate.
See under Phosphoric Acid.

Triethylphosphine (*Phosphorus triethyl*)

$(C_2H_5)_3P$

$C_6H_{15}P$ MW, 118

Colourless liq. with odour of hyacinths. B.p. 127°. D_4^{15} 0·800. n_D^{15} 1·458. Misc. with EtOH, Et_2O. Insol. H_2O.
Add. comp. with CS_2 : red cryst. from Et_2O or EtOH. M.p. 121–2° decomp.
B_2,CuI : plates from ligroin. M.p. 39°.
B,AuCl : needles or prisms. M.p. 80°.
B_2,$PdCl_2$: yellow prisms from Et_2O.

Slotta, Tschesche, *Ber.*, 1927, **60**, 298.

Triethylstibine (*Antimony triethyl*)

$(C_2H_5)_3Sb$

$C_6H_{15}Sb$ MW, 207

Liq. F.p. −98·0°. B.p. 161·4°, 158·5°/730 mm. D^{16} 1·3244. Sol. EtOH, Et_2O. Insol. H_2O. Spontaneously inflammable.

Paneth, Loleit, *J. Chem. Soc.*, 1935, 371.
Dyke, Davies, Jones, *J. Chem. Soc.*, 1930, 465.
Bamford, Levi, Newitt, *J. Chem. Soc.*, 1946, 468.

Triethylurea.
See under Urea.

Trifluoromethane.
See Fluoroform.

ω-Trifluorotoluene.
See Benzotrifluoride.

Triformin (*Glycerol triformate*)

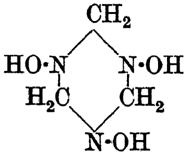
$CH_2·O·CHO$
$CH·O·CHO$
$CH_2·O·CHO$

$C_6H_8O_6$ MW, 176

M.p. 18°. B.p. 266°/762 mm. D^{18} 1·320. n_D^{18} 1·4412. Insol. cold H_2O. Hyd. by hot H_2O.

Romburgh, *Z. physik. Chem.*, 1910, **70**, 459.

Triformoxime (*N-Trihydroxytrimethylene-triamine, trimolecular formaldoxime*)



$C_3H_9O_3N_3$ MW, 135

Amorphous. Insol. H_2O, EtOH, Et_2O. Sol. dil. acids, conc. aq. NaOH. Forms coloured salts with Fe, Ni, Mn. H_2O at 130° ⟶ formaldoxime.

Scholl, *Ber.*, 1891, **24**, 575.

Trigenic Acid.
See Trigonic Acid.

Trigenolline.
See Trigonelline.

Triglycine.
See Diglycylglycine.

Triglycolamidic Acid (*Trimethylamine-1 : 1′ : 1″-tricarboxylic acid*)

$N(CH_2·COOH)_3$

$C_6H_9O_6N$ MW, 191

Prisms from H_2O. Decomp. at 242°. Spar. sol. H_2O.
Tri-Me ester : $C_9H_{15}O_6N$. MW, 233. B.p. 167°/13 mm. D_4^{17} 1·2130. n^{20} 1·4500.
Tri-Et ester : $C_{12}H_{21}O_6N$. MW, 275. Viscous oil. B.p. 193°/18 mm.
Triamide : $C_6H_{12}O_3N_4$. MW, 188. Plates from Alc. NH_3. Decomp. at 205–6°.
Trinitrile : tricyanotrimethylamine. $C_6H_6N_4$. MW, 134. Needles from EtOH. M.p. 125–6°.

Curtius, *J. prakt. Chem.*, 1917, **96**, 232.
Dubsky, Wensink, *Ber.*, 1916, **49**, 1041.

Triglycylglycine (*Glycyl-diglycyl-glycine*)

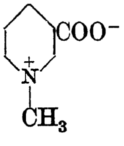
$NH_2·CH_2·CO·[NH·CH_2·CO]_2·NH·CH_2·COOH$

$C_8H_{14}O_5N_4$ MW, 246

Colourless powder. Darkens at 220–70° with evolution of NH_3. Very spar. sol. cold H_2O. Sol. hot H_2O.
Me ester : $C_9H_{16}O_5N_4$. MW, 260. Needles or prisms from MeOH. Darkens at 240°. *Hydrochloride* : plates. M.p. 198–200°.
Et ester : *see* Biuret Base.
Benzoyl : m.p. 235°.

Fischer, *Ber.*, 1904, **37**, 2501.

Trigonelline (*N-Methylnicotinic acid betaine, trigenolline, coffearin, gynesin*)

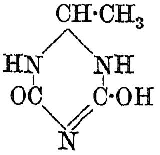


$C_7H_7O_2N$ MW, 137

Constituent of fruit and seeds of species of *Strophanthus*, *Cannabis sativa*, coffee, etc. Prisms + $1H_2O$ from EtOH.Aq. M.p. anhyd. 218° decomp. Very sol. H_2O. Sol. EtOH. Insol. Et_2O, $CHCl_3$. $FeCl_3$ ⟶ red col.
B,HCl : needles or prisms from EtOH. M.p. 245–50° decomp.
B,HI : m.p. 220° decomp.
Picrate : m.p. 205–6°.

Schulze, *Z. physiol. Chem.*, 1909, **60**, 155.
Späth, Bobenberger, *Ber.*, 1944, **77**, 362.

Trigonic Acid (*Trigenic acid, ethylidene biuret*)



$C_4H_7O_2N_3$ MW, 129

Prisms or needles from H_2O. Insol. EtOH.

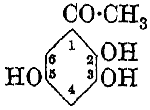
N-*Diacetyl*: m.p. 175–6°. B.p. 200–5°/13 mm.

Picrate: m.p. 211–12° decomp.

Ostrogovich, Ostrogovich, *Chem. Zentr.*, 1936, II, 476.
Liebig, Wöhler, *Ann.*, 1846, **59**, 296.
Ostrogovich, Ostrogovich, *Gazz. chim. ital.*, 1938, **68**, 688.

2 : 3 : 4-Trihydroxyacetophenone.
See Gallacetophenone.

2 : 3 : 5-Trihydroxyacetophenone



$C_8H_8O_4$ MW, 168

Yellow needles from AcOH. M.p. 206–7°. Sol. EtOH, hot AcOH. Spar. sol. hot C_6H_6.

2 : 3 : 5-*Tri-Me ether*: 2 : 3 : 5-trimethoxyacetophenone. $C_{11}H_{14}O_4$. MW, 210. Cryst. from ligroin. M.p. 102–3°.

p-*Nitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 241–2° decomp.

Triacetyl: needles from ligroin. M.p. 106–7°.

Mauthner, *J. prakt. Chem.*, 1933, **136**, 214.

2 : 3 : 6-Trihydroxyacetophenone.

Bright yellow needles from EtOH.Aq. Does not melt. Decomp. above 230°. Aq.NaOH ⟶ yellow col. ⟶ dark greenish-blue col. $FeCl_3$ ⟶ dull yellowish-brown col.

Triacetyl: cryst. from EtOH. M.p. 155°.

Baker, Brown, Scott, *J. Chem. Soc.*, 1939, 1922.

2 : 4 : 5-Trihydroxyacetophenone.

Red needles from H_2O. M.p. 200–2° decomp. Sol. H_2O, hot EtOH, Me_2CO. Insol. C_6H_6, petrol. $FeCl_3$ ⟶ green col. Conc. H_2SO_4 ⟶ yellowish-green col. Ac_2O + AcONa ⟶ 6 : 7-diacetoxy-4-methylcoumarin.

4-*Me ether*: $C_9H_{10}O_4$. MW, 182. Yellow plates from H_2O. M.p. 165–6°. *Diacetyl*: needles from H_2O. M.p. 118–19°.

5-*Me ether*: yellow needles from H_2O. M.p. 166°. $FeCl_3$ ⟶ red col. *Diacetyl*: needles from H_2O. M.p. 127–8°.

4 : 5-*Di-Me ether*: $C_{10}H_{12}O_4$. MW, 196. Yellow needles from H_2O. Cryst. from toluene. M.p. 114–15°. *Oxime*: cryst. from EtOH.Aq. M.p. 162°. *Acetyl*: needles from EtOH.Aq. M.p. 147°.

2 : 4 : 5-*Tri-Me ether*: cryst. from H_2O, EtOH.Aq., or toluene. M.p. 102°. *Semicarbazone*: cryst. from EtOH.Aq. M.p. 206°. *Diacetyl deriv.*: prisms from C_6H_6. M.p. 165–6°.

2 : 4 : 5-*Triacetyl*: cryst. from CCl_4. M.p. 110–11°. *Oxime*: cryst. from EtOH.Aq. M.p. 126–7°. *Semicarbazone*: plates from EtOH. M.p. 186–8°.

Smith, Haller, *J. Am. Chem. Soc.*, 1934, **56**, 237.
Mauthner, *Chem. Abstracts*, 1934, **28**, 3392.

2 : 4 : 6-Trihydroxyacetophenone.
See Phloracetophenone.

3 : 4 : 5-Trihydroxyacetophenone (5-*Acetopyrogallol*).

Needles from H_2O. M.p. 187–8°. Sol. EtOH, Et_2O, AcOH, Me_2CO. Spar. sol. C_6H_6. Insol. pet. ether, ligroin. Aq. $FeCl_3$ ⟶ green col. Alc. $FeCl_3$ ⟶ blue col.

3 : 4 : 5-*Tri-Me ether*: needles from ligroin. M.p. 72° (78°). *Oxime*: needles from H_2O. Prisms from EtOH. M.p. 102–3°. *Semicarbazone*: cryst. from H_2O. M.p. 178–9°.

3 : 4 : 5-*Triacetyl*: needles from ligroin. M.p. 111–12°.

Semicarbazone: needles from EtOH. M.p. 216–17°.

p-*Nitrophenylhydrazone*: red cryst. from AcOH.Aq. Decomp. at 260°.

Mauthner, *J. prakt. Chem.*, 1927, **115**, 137.
Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 523.

α : 3 : 4-Trihydroxyacetophenone.
See 3 : 4-Dihydroxyphenacyl Alcohol.

α : 3 : 4-Trihydroxy-β-aminoethylbenzene.
See Arterenol.

2 : 4 : 6-Trihydroxyanisole.
See Iretol.

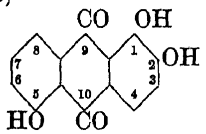
1 : 8 : 9-Trihydroxyanthracene.
See Anthralin.

Trihydroxyanthranol.
See Tetrahydroxyanthracene.

1 : 2 : 3-Trihydroxyanthraquinone.
See Anthragallol.

1 : 2 : 4-Trihydroxyanthraquinone.
See Purpurin.

1 : 2 : 5-Trihydroxyanthraquinone (*Hydroxyanthrarufin*)



$C_{14}H_8O_5$ MW, 256

Red needles from AcOH. M.p. 273–4°. Conc. H_2SO_4 ⟶ violet col.

2-*Me ether*: $C_{15}H_{10}O_5$. MW, 270. Yellow cryst. from EtOH. M.p. 229°.

1 : 2-*Di-Me ether* : $C_{16}H_{12}O_5$. MW, 284. Orange needles from EtOH. M.p. 230·5–231·5°.

1 : 2 : 5-*Tri-Me ether* : $C_{17}H_{14}O_5$. MW, 298. Yellow plates from EtOH. M.p. 203–4°.

1 : 2 : 5-*Triacetyl* : yellow needles from EtOH. M.p. 228–9°.

Puntambecker, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 488.

1 : 2 : 6-Trihydroxyanthraquinone.
See Flavopurpurin.

1 : 2 : 7-Trihydroxyanthraquinone.
See Anthrapurpurin.

1 : 2 : 8-Trihydroxyanthraquinone (*Hydroxychrysazin*).

Red needles from AcOH. M.p. 239–40°. Sublimes. Conc. H_2SO_4 ⟶ reddish-violet col.

2-*Me ether* : $C_{15}H_{10}O_5$. MW, 270. Orange needles from $CHCl_3$–MeOH. M.p. 220°.

2 : 8-*Di-Me ether* : $C_{16}H_{12}O_5$. MW, 284. Brownish-yellow cryst. from $CHCl_3$–MeOH. M.p. 193°.

1 : 2 : 8-*Tri-Me ether* : $C_{17}H_{14}O_5$. MW, 298. Yellow needles from MeOH. M.p. 157°.

1 : 2 : 8-*Triacetyl* : yellow needles. M.p. 224°.

Höchst, D.R.P., 196,980, (*Chem. Zentr.*, 1908, I, 1505).

Graebe, Thode, *Ann.*, 1906, **349**, 221.

1 : 3 : 8-Trihydroxyanthraquinone.

Golden-brown plates from AcOEt. M.p. 287–8°. Conc. H_2SO_4 ⟶ reddish-orange col. Red sols. in alkalis.

Tri-Me ether : greenish-yellow plates from EtOH. M.p. 195–6°.

Triacetyl : pale yellow needles from EtOH. M.p. 194–5°.

Anslow, Breen, Raistrick, *J. Chem. Soc.*, 1940, 427.

1 : 4 : 5-Trihydroxyanthraquinone.

Red needles from $PhNO_2$ or Py.

4-*Acetyl* : yellow needles from AcOH. M.p. 165°.

British Celanese, B.P. 346,355, (*Chem. Abstracts*, 1932, **26**, 1948).

1 : 4 : 6-Trihydroxyanthraquinone.

Reddish-brown powder. Does not melt below 300°. Spar. sol. hot H_2O. Sol. alkalis ⟶ violet col. $Pb(OAc)_4$ ⟶ 1 : 4 : 9 : 10-diquinone.

6-*Acetyl* : cryst. from AcOH.

Crossley, *J. Am. Chem. Soc.*, 1918, **40**, 404.

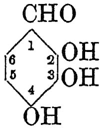
5 : 7 : 8-Trihydroxyanthraquinone-1-carboxylic Acid.
See Boletol.

1 : 3 : 4 - Trihydroxyanthraquinone - 2 - carboxylic Acid.
See Purpurin-3-carboxylic Acid.

4 : 5 : 7 - Trihydroxyanthraquinone - 2 - carboxylic Acid.
See Emodic Acid.

Trihydroxyanthrone.
See Tetrahydroxyanthracene.

2 : 3 : 4-Trihydroxybenzaldehyde



$C_7H_6O_4$ MW, 154

Needles from H_2O. M.p. 161–2°.

3 : 4-*Di-Me ether* : $C_9H_{10}O_4$. MW, 182. Needles from H_2O, prisms from petrol. M.p. 74°. *Phenylhydrazone* : yellow prisms from MeOH. M.p. 156°.

2 : 3 : 4-*Tri-Me ether* : 2 : 3 : 4-trimethoxybenzaldehyde. $C_{10}H_{12}O_4$. MW, 196. Prisms from petrol. M.p. 37° (30°). *Phenylhydrazone* : yellow prisms from MeOH. M.p. 155–6°.

2 : 3 : 4-*Tri-Et ether* : 2 : 3 : 4-triethoxybenzaldehyde. $C_{13}H_{18}O_4$. MW, 238. Cryst. M.p. 70°.

Oxime : needles from H_2O. M.p. 204° decomp.

Baker, Smith, *J. Chem. Soc.*, 1931, 2544.

Schaaf, Labouchère, *Helv. Chim. Acta*, 1924, **7**, 357.

Barger, Ewins, *J. Chem. Soc.*, 1910, **97**, 2258.

Gattermann, Berchelmann, *Ber.*, 1898, **31**, 1768.

2 : 3 : 5-Trihydroxybenzaldehyde.

2 : 3-*Di-Me ether* : cryst. from H_2O. M.p. 152°.

2 : 3 : 5-*Tri-Me ether* : 2 : 3 : 5-trimethoxybenzaldehyde. Cryst. from EtOH.Aq. M.p. 71°.

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, **53**, 3074.

2 : 4 : 5-Trihydroxybenzaldehyde.

Cryst. from H_2O. M.p. 223°. $FeCl_3$ ⟶ green col.

4-*Me ether* : 2 : 5-dihydroxyanisaldehyde. $C_8H_8O_4$. MW, 168. Prisms from EtOH or AcOH. M.p. 209°. Spar. sol. hot EtOH, AcOH, Me_2CO. $FeCl_3$ ⟶ bluish-green col.

4 : 5-*Di-Me ether* : 6-hydroxyveratric aldehyde. $C_9H_{10}O_4$. MW, 182. Needles from H_2O. Plates from EtOH.Aq. M.p. 105°. $FeCl_3$ ⟶ green col.

2 : 4 : 5-*Tri-Me ether* : *see* Asarylaldehyde.

4-*Me*-5-*Et ether* : $C_{10}H_{12}O_4$. MW, 196. Prisms from MeOH. M.p. 112–13°. Sol. EtOH, Me_2CO. Spar. sol. H_2O. $FeCl_3$ ⟶ green col.

5-*Me*-4-*Et ether* : prisms from MeOH. M.p. 91°. *Phenylhydrazone* : m.p. 157–8°.

2 : 4-*Di-Me*-5-*Et ether* : $C_{11}H_{14}O_4$. MW, 210. Needles from MeOH. M.p. 110°.

2 : 4 : 5-*Tri-Et ether* : 2 : 4 : 5-triethoxybenzaldehyde. $C_{13}H_{18}O_4$. MW, 238. Cryst. from EtOH. M.p. 95°.

4-*Benzoyl* : plates from EtOH.Aq. M.p. 184°.

2 : 4 : 5-*Triacetyl* : prisms from EtOH. M.p. 115°.

Head, Robertson, *J. Chem. Soc.*, 1930, 2436.

Gattermann, Köbner, *Ber.*, 1899, **32**, 282.

2 : 4 : 6-Trihydroxybenzaldehyde (*Phloroglucinaldehyde*).

Needles + $2H_2O$ from H_2O. Darkens on heating. $FeCl_3$ ⟶ red col.

2-*Me ether* : needles from EtOH. M.p. 200–2°. 4 : 6-*Diacetyl* : plates from ligroin. M.p. 107°.

4-*Me ether* : 2-*benzoyl*, plates from EtOH. M.p. 109°.

4 : 6-*Di-Me ether* : needles or plates from MeOH. M.p. 70–1°. 2-*Benzoyl* : needles from AcOEt. M.p. 148°.

2 : 4 : 6-*Tri-Me ether* : 2 : 4 : 6-trimethoxybenzaldehyde. Needles from EtOH. M.p. 118°. *Oxime* : needles from MeOH. M.p. 201–3°.

Oxime : cryst. + $1H_2O$ from H_2O. Decomp. at 195°.

Diacetyl deriv. : needles from EtOH.Aq. M.p. 102–3°.

Triacetyl : prisms from EtOH. M.p. 156–7° (151°).

2-*Benzoyl* : prisms from $CHCl_3$. M.p. 198–200°.

Malkin, Nierenstein, *J. Am. Chem. Soc.*, 1931, **53**, 241.

Robinson *et al.*, *J. Chem. Soc.*, 1930, 804; 1928, 1457; 1927, 1712; 1925, 1184.

Gattermann, Köbner, *Ber.*, 1899, **32**, 280.

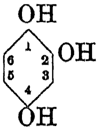
3 : 4 : 5-Trihydroxybenzaldehyde.

See Gallaldehyde.

1 : 2 : 3-Trihydroxybenzene.

See Pyrogallol.

1 : 2 : 4-Trihydroxybenzene (*Hydroxyhydroquinone, hydroxyquinol*)



$C_6H_6O_3$ MW, 126

Plates from Et_2O. M.p. 140·5°. Sol. H_2O, EtOH, Et_2O. Insol. $CHCl_3$, CS_2, C_6H_6. Oxidises in air. Conc. H_2SO_4 ⟶ green col. ⟶ red on warming. $FeCl_3$ + NaOH ⟶ red col. Ag_2O ⟶ 2-hydroxy-*p*-benzoquinone.

Picrate : orange-red needles. M.p. 96°.

1-*Me ether* : $C_7H_8O_3$. MW, 140. Prisms from C_6H_6. M.p. 66–7°. 2 : 4-*Diacetyl* : prisms from MeOH. M.p. 62–4°.

2-*Me ether* : plates from H_2O. Prisms from C_6H_6. M.p. 84°. 1 : 4-*Diacetyl* : needles from MeOH. M.p. 93–4°.

1 : 2 : 4-*Tri-Me ether* : $C_9H_{12}O_3$. MW, 168. Liq. B.p. 247°.

1 : 2-*Methylene ether* : *see* Sesamol.

2-*Et ether* : $C_8H_{10}O_3$. MW, 154. Prisms from EtOH. M.p. 112·5°.

1 : 2-*Di-Et ether* : $C_{10}H_{14}O_3$. MW, 182. Cryst. from C_6H_6 or EtOH. M.p. 65–7°.

1 : 2 : 4-*Tri-Et ether* : $C_{12}H_{18}O_3$. MW, 210. Needles from EtOH.Aq. M.p. 34°.

1 : 2 : 4-*Triacetyl* : cryst. from EtOH. M.p. 96–7°.

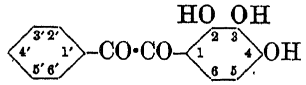
Healey, Robinson, *J. Chem. Soc.*, 1934, 1626.

Vliet, *Organic Syntheses*, Collective Vol. I, 310.

1 : 3 : 5-Trihydroxybenzene.

See Phloroglucinol.

2 : 3 : 4-Trihydroxybenzil



$C_{14}H_{10}O_5$ MW, 258

Needles from H_2O. M.p. 143°. Sol. H_2O, EtOH, Et_2O, C_6H_6.

Oxime : yellow cryst. M.p. 144°.

Dioxime : cryst. from EtOH. M.p. 168°.

Noelting, Kadiera, *Ber.*, 1906, **39**, 2059.

2 : 4 : 6-Trihydroxybenzil.

Colourless needles from EtOH.Aq. Darkens at 260°, m.p. 287°. $FeCl_3$ ⟶ purple col. Reduces Fehling's.

Triacetyl : m.p. 248°.

Marsh, Stephen, *J. Chem. Soc.*, 1925, 1636.

2 : 4 : 2′-Trihydroxybenzil.

2′-*Me ether* : $C_{15}H_{12}O_5$. MW, 272. Darkens at 210°, m.p. 223°. 2 : 4-*Diacetyl* : m.p. 144°.

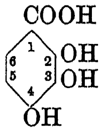
Marsh, Stephen, *J. Chem. Soc.*, 1925, 1636.

2 : 4 : 4′-Trihydroxybenzil.

4′-*Me ether* : darkens at 225°, m.p. 234°. 2 : 4-*Diacetyl* : m.p. 178·5°.

Marsh, Stephen, *J. Chem. Soc.*, 1925, 1636.

2 : 3 : 4-Trihydroxybenzoic Acid (*Pyrogallol-4-carboxylic acid*)



$C_7H_6O_5$ MW, 170

Needles + H_2O from H_2O. Decomp. at 207–8° (215–20°). Sol. EtOH. Spar. sol. Et_2O. Sublimes in CO_2. $FeCl_3$ ⟶ violet col. Hot H_2O ⟶ pyrogallol.

Me ester : $C_8H_8O_5$. MW, 184. Needles + $2\frac{1}{2}H_2O$ from H_2O. M.p. 151–2°. 4-*Me-ether* : $C_9H_{10}O_5$. MW, 198. Needles from EtOH.Aq. M.p. 101–4°. 4-*Me ether*-2 : 3-*diacetyl* : plates from MeOH. M.p. 108°. 3 : 4-*Di-Me ether* : $C_{10}H_{12}O_5$. MW, 212. Needles or prisms from EtOH. M.p. 75–8°. 3 : 4-*Di-Me ether*-2-*acetyl* :

cryst. from EtOH. M.p. 62–4°. 2 : 3-*Di-Me ether*-4-*benzoyl*: cryst. from EtOH.Aq. M.p. 79–80°.

Et ester: $C_9H_{10}O_5$. MW, 198. Cryst. + $1H_2O$ from H_2O. M.p. anhyd. 102°.

Hydrazide: grey cryst. M.p. above 180° decomp.

4-*Me ether*: $C_8H_8O_5$. MW, 184. Needles from H_2O. M.p. 207–8°. $FeCl_3$ ⟶ blue col.

2 : 3-*Di-Me ether*: $C_9H_{10}O_5$. MW, 198. Plates from H_2O. M.p. 154–5°. Insol. ligroin. Sol. MeOH, AcOEt, Me_2CO. $FeCl_3$ ⟶ brownish-yellow col.

3 : 4-*Di-Me ether*: needles from H_2O. M.p. 169–72°.

2 : 3 : 4-*Tri-Me ether*: $C_{10}H_{12}O_5$. MW, 212. Cryst. from H_2O or petrol. M.p. 99°. *Me ester*: $C_{11}H_{14}O_5$. MW, 226. Oil. B.p. 281°. *Chloride*: $C_{10}H_{11}O_4Cl$. MW, 230·5. Cryst. from petrol. M.p. 42°. B.p. 175°/11 mm. *Amide*: $C_{10}H_{13}O_4N$. MW, 211. M.p. 130–1°.

2 : 3 : 4-*Tri-Et ether*: $C_{13}H_{18}O_5$. MW, 254. Cryst. from EtOH. M.p. 105°.

2 : 3-*Diacetyl*: needles + $1H_2O$ from MeOH.Aq. M.p. anhyd. 157°.

2 : 3 : 4-*Triacetyl*: prisms from xylene. M.p. 164°.

4-*Benzoyl*: plates from MeOH.Aq. M.p. 210–11°.

2 : 3-*Diacetyl-4-benzoyl*: needles from petrol-$CHCl_3$. M.p. 161–2°.

Hemmelmayr, *Monatsh.*, 1917, **38**, 81, 88.
Pacsu, *Ber.*, 1923, **56**, 418.
Kostanecki, *Ber.*, 1885, **18**, 3205.

2 : 3 : 5-Trihydroxybenzoic Acid.

2 : 3-*Di-Me ether*: cryst. M.p. 186–8°.

2 : 3 : 5-*Tri-Me ether*: plates from H_2O. M.p. 105° (141–3°).

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, **53**, 3074.
Faltis, Kloiber, *Monatsh.*, 1929, **53** and **54**, 633.

2 : 3 : 6-Trihydroxybenzoic Acid.

2 : 3-*Di-Me ether*: needles from H_2O. M.p. 82°.

2 : 3 : 6-*Tri-Me ether*: cryst. from H_2O. M.p. 145–6°.

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, **53**, 3075.

2 : 4 : 5-Trihydroxybenzoic Acid (*Hydroxyhydroquinone-carboxylic acid*).

Needles + $\frac{1}{2}H_2O$ from H_2O. M.p. 217–18° decomp. $FeCl_3$ ⟶ blue to red col. Loses CO_2 with H_2O at 100°.

4 : 5-*Di-Me ether*: $C_9H_{10}O_5$. MW, 198. Brown needles from H_2O. M.p. 202° decomp. *Me ester*: $C_{10}H_{12}O_5$. MW, 212. Needles from H_2O. M.p. 95°.

2 : 4 : 5-*Tri-Me ether*: *see* Asarylic Acid.

2 : 4-*Di-Me-5-Et ether*: $C_{11}H_{14}O_5$. MW, 226. Needles from H_2O, prisms from C_6H_6. M.p. 137°.

2 : 5-*Di-Me-4-Et ether*: prisms from H_2O. M.p. 130°.

2 : 4 : 5-*Tri-Et ether*: $C_{13}H_{18}O_5$. MW, 254. Needles from EtOH.Aq. M.p. 134°.

2 : 4 : 5-*Triacetyl*: plates or needles from C_6H_6. M.p. 162–3°.

Head, Robertson, *J. Chem. Soc.*, 1930, 2439.

2 : 4 : 6-Trihydroxybenzoic Acid (*Phloroglucinol-carboxylic acid*).

Cryst. + $1H_2O$. Decomp. on heating. $FeCl_3$ ⟶ blue col. Absorbs O in alk. sol.

Me ester: $C_8H_8O_5$. MW, 184. Cryst. from EtOH.Aq. M.p. 174–6°. 4-*Me-2-Et ether*: $C_{11}H_{14}O_5$. MW, 226. Needles from EtOH. M.p. 97–9°. 2 : 4 : 6-*Tri-acetyl*: needles from MeOH. M.p. 77–9°.

Et ester: $C_9H_{10}O_5$. MW, 198. Prisms or needles + $1H_2O$ from H_2O, prisms from ligroin. M.p. 129°. $FeCl_3$ ⟶ violet col. 2 : 6-*Di-Et ether*: $C_{13}H_{18}O_5$. MW, 254. Prisms from MeOH. M.p. 180–1°.

4-*Me ether*: $C_8H_8O_5$. MW, 184. Grey needles from Et_2O–C_6H_6. M.p. 141° decomp. *Me ester*: $C_9H_{10}O_5$. MW, 198. Needles from MeOH. M.p. 114–16°.

2 : 6-*Di-Me ether*: $C_9H_{10}O_5$. MW, 198. Plates. M.p. 175° decomp. *Me ester*: $C_{10}H_{12}O_5$. MW, 212. Plates from MeOH.Aq. M.p. 189° decomp.

4 : 6-*Di-Me ether*: needles from Et_2O–C_6H_6. M.p. 152–4° decomp. *Me ester*: needles from MeOH. M.p. 107–9°.

2 : 4 : 6-*Tri-Me ether*: $C_{10}H_{12}O_5$. MW, 212. Needles from EtOH.Aq. M.p. 142–4° decomp. *Me ester*: $C_{11}H_{14}O_5$. MW, 226. Cryst. from MeOH. M.p. 67–70°. *Et ester*: $C_{12}H_{16}O_5$. MW, 240. Needles from ligroin. M.p. 77–8°.

4-*Me-2 : 6-Di-Et ether*: $C_{12}H_{16}O_5$. MW, 240. Plates from MeOH. M.p. 168°.

4-*Acetyl*: needles from MeOH. M.p. 177–8°.

Skraup, *Monatsh.*, 1889, **10**, 724.
Sonn, Winzer, *Ber.*, 1928, **61**, 2303.

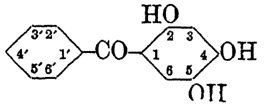
3 : 4 : 5-Trihydroxybenzoic Acid.

See Gallic Acid.

2 : 3 : 4-Trihydroxybenzophenone.

See Gallobenzophenone.

2 : 4 : 5-Trihydroxybenzophenone



$C_{13}H_{10}O_4$ MW, 230

5-*Me ether*: $C_{14}H_{12}O_4$. MW, 244. Yellow needles from EtOH. M.p. 183–5°.

4 : 5-*Di-Me ether*: $C_{15}H_{14}O_4$. MW, 258. Greenish-yellow cryst. from EtOH.Aq. M.p. 106–7°. 2-*Acetyl*: yellow needles from EtOH.Aq. M.p. 108–10°.

2 : 4 : 5-*Tri-Me ether* : $C_{16}H_{16}O_4$. MW, 272. Yellow needles from H_2O. M.p. 97°. *Phenylhydrazone* : leaflets. M.p. 178–9°.

Bargellini, Martegiani, *Atti accad. Lincei*, 1911, **20**, 184.

2 : 4 : 6-Trihydroxybenzophenone.
See Phlorbenzophenone.

2 : 3 : 4′-Trihydroxybenzophenone.
Yellow needles from MeOH.Aq. M.p. 169°. $FeCl_3 \longrightarrow$ brownish-green col.
Tri-Me ether : $C_{16}H_{16}O_4$. MW, 272. Cryst. from MeOH.Aq. M.p. 86°.

Baker, Smith, *J. Chem. Soc.*, 1936, 348.

2 : 4 : 4′-Trihydroxybenzophenone.
Yellow needles from H_2O. M.p. 200°. $FeCl_3 \longrightarrow$ purple col.
4′-*Me ether* : $C_{14}H_{12}O_4$. MW, 244. Needles from H_2O. M.p. 165°. 2 : 4-*Diacetyl* : needles from EtOH.Aq. M.p. 128–30°.
Tri-Me ether : needles from EtOH. M.p. 73–4°.
Triacetyl : needles from H_2O. M.p. 96–8°.

Komarowski, Kostanecki, *Ber.*, 1894, **27**, 1999.

2 : 6 : 2′-Trihydroxybenzophenone.
Yellow cryst. from EtOH.Aq. M.p. 133–4°. Sol. hot H_2O. NaOH $\longrightarrow$ yellow col.

Michael, *Am. Chem. J.*, 1883, **5**, 89.

3 : 4 : 5-Trihydroxybenzophenone.
Yellow plates + $1H_2O$, colourless anhyd. cryst. from $CHCl_3$. M.p. anhyd. 177–8°. Very sol. hot H_2O. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. C_6H_6, petrol.

Fischer, *Ber.*, 1909, **42**, 1018.

3 : 4 : 3′-Trihydroxybenzophenone.
Tri-Me ether : $C_{16}H_{16}O_4$. MW, 272. Needles from MeOH. M.p. 83–4°. *Oxime* : prisms from EtOH. M.p. 128°.

Lea, Robinson, *J. Chem. Soc.*, 1926, 2355.

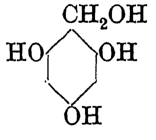
3 : 4 : 4′-Trihydroxybenzophenone.
Tri-Me ether : needles from EtOH. M.p. 98–9°.

Kostanecki, Tambor, *Ber.*, 1906, **39**, 4026.

3 : 5 : 4′-Trihydroxybenzophenone.
Tri-Me ether : needles from C_6H_6. M.p. 97–8°.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 406.

2 : 4 : 6-Trihydroxybenzyl Alcohol



$C_7H_8O_4$ MW, 156

2 : 4 : 6-*Tri-Me ether* : $C_{10}H_{14}O_4$. MW, 198. Cryst. from pet. ether. M.p. 63°.

Freudenberg, Harder, *Ann.*, 1927, **451**, 222.

3 : 4 : 5-Trihydroxybenzyl Alcohol.
See Gallyl Alcohol.

3 : 4 : 5-Trihydroxybenzylamine.
See Gallylamine.

1 : 2 : 3-Trihydroxybutane.
See 1-Methylglycerol.

1 : 2 : 4-Trihydroxybutane (1 : 2 : 4-*Butantriol*)

$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2OH$

$C_4H_{10}O_3$ MW, 106

Hygroscopic syrup with sweet, burning taste. B.p. 179°/13 mm. D^{20} 1·18. n_D^{20} 1·47.
4-*Me ether* : $C_5H_{12}O_3$. MW, 120. B.p. 121°/12 mm. D^0 1·11. n_D^{20} 1·448. *Di-phenylurethane* : m.p. 111–12°.
1-*Et ether* : $C_6H_{14}O_3$. MW, 134. Slightly yellow oil. B.p. 210°.
4-*Et ether* : b.p. 130°/14 mm. D^0 1·08. n_D^{15} 1·45. *Di-phenylurethane* : m.p. 98–9°.
Tri-phenylurethane : needles from C_6H_6. M.p. 149–52°.
1 : 4-*Diacetyl* : b.p. 161–3°/18 mm. D_0^{16} 1·15. n_D^{16} 1·446.
Triacetyl : b.p. 150°/11 mm. D_0^{19} 1·13. n_D^{19} 1·436.

Pariselle, *Ann. chim.*, 1911, **24**, 346.
Wagner, *Ber.*, 1894, **27**, 2437.

1 : 3 : 3-Trihydroxybutane (1 : 3 : 3-*Butantriol*)

$$HO{\cdot}CH_2{\cdot}CH_2{\cdot}\overset{\displaystyle OH}{\underset{\displaystyle OH}{C}}{\cdot}CH_3$$

$C_4H_{10}O_3$ MW, 106

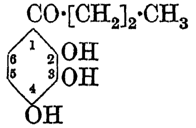
Tri-Me-ether : $C_7H_{16}O_3$. MW, 148. B.p. 63°/20 mm. D_4^{20} 0·9398. n_D^{20} 1·4112.
Tri-Et ether : $C_{10}H_{22}O_3$. MW, 192. B.p. 75°/9 mm. D_4^{20} 0·8940. n_D^{20} 1·4148.
Tributyl ether : $C_{16}H_{34}O_2$. MW, 276. B.p. 120°/3 mm. D_4^{20} 0·8745. n_D^{20} 1·4310.

Dykstra, *J. Am. Chem. Soc.*, 1935, **57**, 2257.

1 : 2 : 3-Trihydroxybutyraldehyde.
See Erythrose *and* Threose.

Trihydroxybutyric Acid.
See Erythronic Acid *and* Threonic Acid.

2 : 3 : 4-Trihydroxybutyrophenone (4-*Butyrylpyrogallol*)



$C_{10}H_{12}O_4$ MW, 196

Yellow needles. M.p. anhyd. 100°.

Badische, D.R.Ps., 49,149, 50,451.

2 : 4 : 6-Trihydroxybutyrophenone.
See Phlorbutyrophenone.

3 : 4 : 5-Trihydroxybutyrophenone (*5-Butyrylpyrogallol*).

Tri-Me ether: $C_{13}H_{18}O_4$. MW, 238. Needles from MeOH. M.p. 51–52·5°. Sol. common org. solvents. p-*Nitrophenylhydrazone*: m.p. 160°.

Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 526.

2 : 3 : 4-Trihydroxycaproic Acid (*Digitoxic acid*)

$CH_3·CH(OH)·CH(OH)·CH(OH)·CH_2·COOH$

$C_6H_{12}O_5$ MW, 164

Colourless syrup. $HNO_3 \longrightarrow$ 1 : 2-dihydroxyglutaric acid.

Quinine salt: needles or columns. M.p. 164°. Very sol. MeOH.Aq. Spar. sol. cold H_2O.

Brucine salt: columns from EtOH–Et_2O. M.p. 124°. Very sol. H_2O. Mod. sol. EtOH.

Phenylhydrazide: cryst. from EtOH. M.p. 159°.

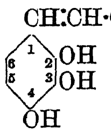
Zemplén, *Ber.*, 1923, **56**, 688.

Kiliani, *Arch. pharm.*, 1913, **251**, 579.

2 : 3 : 4-Trihydroxycaproic Aldehyde.

See Digitoxose.

2 : 3 : 4-Trihydroxycinnamic Acid (*Daphnetic acid*)



$C_9H_8O_5$ MW, 196

Tri-Me ether: $C_{12}H_{14}O_5$. MW, 238. Needles from EtOH. M.p. 172°.

4-*Me*-2 : 3-*Di-Et-ether*: $C_{14}H_{18}O_5$. MW, 266. Cryst. from pet. ether. M.p. 157–8°.

Tri-Et ether: $C_{15}H_{20}O_5$. MW, 280. Cryst. M.p. 193°. Very sol. hot EtOH, Et_2O, C_6H_6. Insol. H_2O, CS_2. Ox. $\longrightarrow$ 2 : 3 : 4-trimethoxybenzaldehyde and 2 : 3 : 4-trimethoxybenzoic acid.

Will, Jung, *Ber.*, 1884, **17**, 1086.

2 : 4 : 5-Trihydroxycinnamic Acid (*Aesculetic acid*).

Me ester: $C_{10}H_{10}O_5$. MW, 210. Cryst. M.p. 109°.

5-*Me ether*: $C_{10}H_{10}O_5$. MW, 210. Pale yellow needles + $1H_2O$ from H_2O. M.p. 178–80° decomp. Spar. sol. Et_2O. Boiling dil. acids eliminates CO_2.

Tri-Me ether: $C_{12}H_{14}O_5$. MW, 238. Needles from EtOH.Aq. M.p. 169°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. cold H_2O. *Me ester*: $C_{13}H_{16}O_5$. MW, 252. Prisms from EtOH.Aq. M.p. 109°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O.

Tri-Et ether: $C_{15}H_{20}O_5$. MW, 280. Two forms. (i) Cryst. from EtOH. M.p. 102–3°. Conc. HCl $\longrightarrow$ (ii). Ox. $\longrightarrow$ triethoxybenzaldehyde and triethoxybenzoic acid. *Et ester*: $C_{17}H_{24}O_5$. MW, 308. Pale yellow prisms. M.p. 51°. (ii) Cryst. from EtOH.Aq. M.p. 144°. Dist. with slight decomp. Sol. EtOH, Et_2O, C_6H_6. Very spar. sol. H_2O. *Et ester*: plates from EtOH. M.p. 75°.

Will, *Ber.*, 1883, **16**, 2109.

Tiemann, Will, *Ber.*, 1882, **15**, 2082.

2 : 4 : 6-Trihydroxycinnamic Acid.

Tri-Me ether: $C_{12}H_{14}O_5$. MW, 238. Yellowish-white needles. M.p. 218° decomp. Very sol. MeOH. Spar. sol. H_2O, Et_2O. *Me ester*: $C_{13}H_{16}O_5$. MW, 252. Needles. M.p. 134–5°.

Herzig, Wenzel, Gehringer, *Monatsh.*, 1903, **24**, 868.

3 : 4 : 5-Trihydroxycinnamic Acid.

Needles + $1H_2O$ from H_2O. Anhyd. at 120°. M.p. 207–8° decomp. Very sol. EtOH, Me_2CO, hot H_2O. Spar. sol. cold H_2O, Et_2O, AcOH. Insol. $CHCl_3$, C_6H_6, CS_2, pet. ether. Sol. conc. $H_2SO_4 \longrightarrow$ yellow sol. Reduces cold $NH_3.AgNO_3$, and Fehling's on warming. KCN sol. $\longrightarrow$ red $\longrightarrow$ violet $\longrightarrow$ yellowish-green col.

3 : 5-*Di-Me ether*: *see* Sinapic Acid.

Tri-Me ether: *see under* Sinapic Acid.

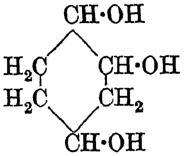
Triacetyl: needles from $CHCl_3$–petrol. M.p. 168°.

Rosenmund, Boehm, *Ann.*, 1924, **437**, 144.

1 : 2 : 3-Trihydroxycyclohexane.

See Pyrogallitol.

1 : 2 : 4-Trihydroxycyclohexane



$C_6H_{12}O_3$ MW, 132

Cryst. from EtOH–Et_2O. M.p. 122°.

Zelinsky, Titowa, *Ber.*, 1931, **64**, 140.

1 : 3 : 5-Trihydroxycyclohexane.

See Phloroglucitol.

Trihydroxydiacetylbenzene.

See Gallodiacetophenone.

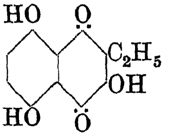
5 : 7 : 4′-Trihydroxy-3 : 5′-dimethoxyflavonol.

See Syringetin.

1 : 1 : 1-Trihydroxyethane.

See Orthoacetic Acid.

3 : 5 : 8-Trihydroxy-2-ethyl-1 : 4-naphthoquinone



$C_{12}H_{10}O_5$ MW, 234

Pigment from the ovaries of *Arbacia*. Red rods from 50% EtOH.Aq. or ligroin. M.p. 187°. KOH $\longrightarrow$ red col.

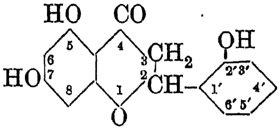
3-*Me ether*: red cryst. from Et_2O. M.p. 83–4°.

5 : 8-*Di-Me ether*: orange-red cryst. from Et_2O. M.p. 156°.

Kuroda, *Chem. Abstracts*, 1940, **34**, 6215.
Wallenfels, *Ber.*, 1942, **75**, 785.
Brunner, Singule, *Monatsh.*, 1947, **77**, 251.

7 : 3′ : 4′-Trihydroxyflavanol.
See Fustin.

5 : 7 : 2′-Trihydroxyflavanone



$C_{15}H_{12}O_5$ MW, 272

Needles from AcOH.Aq. M.p. 185–7°. $FeCl_3$ ⟶ red col.

2′-*Me ether*: *see* Citronetin.

5(7)-*Me ether*: $C_{16}H_{14}O_5$. MW, 286. Cryst. from EtOH. M.p. 192°. $FeCl_3$ ⟶ reddish-brown col.

Tri-Me ether: $C_{18}H_{18}O_5$. MW, 314. Needles from EtOH. M.p. 124–5°. Sol. conc. H_2SO_4 ⟶ yellowish-brown col.

Shinoda, Sato, *Chem. Zentr.*, 1931, II, 2326.

5 : 7 : 3′-Trihydroxyflavanone.

Plates from AcOH. M.p. 240–1°. $FeCl_3$ ⟶ violet-brown col.

3′-*Me ether*: $C_{16}H_{14}O_5$. MW, 286. Plates. M.p. 179–80°. *Oxime*: plates from EtOH.Aq. M.p. 194–5°. 5(7)-*Me ether*: $C_{17}H_{16}O_5$. MW, 300. Plates from EtOH. M.p. 96°. *Diacetyl*: cryst. from EtOH. M.p. 106–7°.

5(7)-*Me ether*: needles from EtOH. M.p. 182°.

Acetyl: cryst. from C_6H_6–pet. ether. M.p. 43–5°.

Shinoda, Sato, *Chem. Zentr.*, 1931, II, 2326.

5 : 7 : 4′-Trihydroxyflavanone.
See Naringenin.

7 : 8 : 2′-Trihydroxyflavanone.

Colourless cryst. M.p. 215–16°.

Triacetyl: m.p. 119°.

Geissman, Clinton, *J. Am. Chem. Soc.*, 1946, **68**, 697.

7 : 8 : 4′-Trihydroxyflavanone.

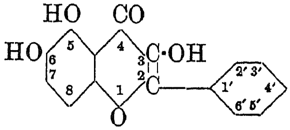
Colourless cryst. M.p. 194°.

Triacetyl: m.p. 165–6°.

Geissman, Clinton, *J. Am. Chem. Soc.*, 1946, **68**, 697.

7 : 3′ : 4′-Trihydroxyflavanone.
See Butin.

3 : 5 : 6-Trihydroxyflavone (5 : 6-*Dihydroxyflavonol*)



$C_{15}H_{10}O_5$ MW, 270

M.p. 183–5°.

Tri-Me ether: m.p. 130–2°.

Rajagopalan, Row, Seshadri, *Proc. Indian Acad. Sci.*, 1946, **23**, 97, (*Chem. Abstracts*, 1947, **41**, 120).

3 : 5 : 7-Trihydroxyflavone.
See Galangin.

3 : 6 : 7-Trihydroxyflavone (6 : 7-*Dihydroxyflavonol*).

Prisms. M.p. 312–15°. $FeCl_3$ ⟶ dark greenish-brown col.

Triacetyl: m.p. 191–2°.

Rao, Row, Seshadri, *Proc. Indian Acad. Sci.*, 1945, **22**, 297, (*Chem. Abstracts*, 1946, **40**, 3756).

3 : 7 : 8-Trihydroxyflavone (7 : 8-*Dihydroxyflavonol*).

M.p. 248–9°.

Narasimhachari, Row, Seshadri, *Chem. Abstracts*, 1949, **43**, 640.

3 : 5 : 4′-Trihydroxyflavone (5 : 4′-*Dihydroxyflavonol*).

M.p. 214–15°.

Tri-Me ether: m.p. 141–2°.

Triacetyl: m.p. 179–80°.

Seshadri, Venkateswarlu, *Chem. Abstracts*, 1949, **43**, 1407.

3 : 3′ : 4′-Trihydroxyflavone (3′ : 4′-*Dihydroxyflavonol*).

Free flavone unknown.

4′-β-α-*Glucoside*: pale yellow needles + $1H_2O$. Foams at 140°, resolidifies at 150° and melts at 232–6°. $[\alpha]_D^{20}$ − 48° in dioxan.

Reichel, Marchand, *Ber.*, 1943, **76**, 1132.

5 : 6 : 7-Trihydroxyflavone.
See Baicalein.

5 : 7 : 8-Trihydroxyflavone.
See Norwogonin.

5 : 6 : 4′-Trihydroxyflavone.

Yellow needles. M.p. 298°. NaOH.Aq. ⟶ orange-yellow col. Alc. $FeCl_3$ ⟶ apple green col.

Tri-Me ether: needles from EtOH. M.p. 164°.

Triacetyl: needles from EtOH. M.p. 209°.

Baker, Simmonds, *J. Chem. Soc.*, 1940, 1370.

5 : 7 : 4′-Trihydroxyflavone.
See Apigenin.

6 : 7 : 8-Trihydroxyflavone.

Yellow cryst. from AcOH or EtOH.Aq. M.p. 280°.

Tri-Me ether: yellow cryst. from MeOH. M.p. 146°.

Triacetyl: colourless cryst. from AcOH or EtOH.Aq. M.p. 197–8°.

Bargellini, Oliverio, *Ber.*, 1942, **75**, 2086.

6 : 7 : 4′-Trihydroxyflavone.

Yellow needles + $1H_2O$ from AcOH.Aq. Anhyd. at 110°. Decomp. at 300°. Sol. EtOH, NaOH.Aq. Alc. $FeCl_3$ ⟶ green col. Conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Di-Me ether: $C_{17}H_{14}O_5$. MW, 298. Cryst. + H_2O. M.p. 158–62° decomp. Sol. EtOH, AcOH. Spar. sol. C_6H_6. Alc. $FeCl_3$ ⟶ red col. *Acetyl*: cryst. from EtOH. M.p. 164–6°.

Tri-Me ether: $C_{18}H_{16}O_5$. MW, 312. Needles from EtOH. M.p. 184–6°. Sol. AcOH, C_6H_6. Fluor. sol. in EtOH. Sol. conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Triacetyl: needles from AcOH. M.p. 234–6°.

Bargellini, Grippa, *Gazz. chim. ital.*, 1927, **57**, 605.

7 : 8 : 4′-Trihydroxyflavone.

Yellow needles from EtOH.Aq. Sinters at 279°, m.p. 299–300° decomp. Conc. H_2SO_4 ⟶ yellow sol. Alc. $FeCl_3$ ⟶ green col. NaOH.Aq. ⟶ orange sol.

Tri-Me ether: yellow needles from EtOH. M.p. 189–90°.

Triacetyl: white needles. M.p. 183°.

Badhwar, Kang, Venkataraman, *J. Chem. Soc.*, 1932, 1109.

7 : 3′ : 4′-Trihydroxyflavone.

3′ : 4′-*Di-Me ether*: $C_{17}H_{14}O_5$. MW, 298. Plates from AcOH.Aq. M.p. 255°. Sol. EtOH with violet fluor. No col. with $FeCl_3$.

Baker, *J. Chem. Soc.*, 1933, 1387.

3′ : 4′ : 5′-Trihydroxyflavone.

Pale yellow needles from EtOH.Aq. Does not melt below 280°. Conc. H_2SO_4 ⟶ yellow sol. NaOH ⟶ red sol. $FeCl_3$ ⟶ green ppt.

Tri-Me ether: $C_{18}H_{16}O_5$. MW, 312. Needles from EtOH.Aq. M.p. 174–5°.

Triacetyl: prisms from EtOH. M.p. 195–6°.

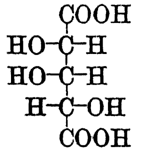
Hattori, *Chem. Zentr.*, 1932, II, 710.

Trihydroxyflavonol.

See Tetrahydroxyflavone.

Trihydroxyglutaric Acid

I. Arabo-trihydroxyglutaric Acid.



$C_5H_8O_7$ MW, 180

d-.

Plates from H_2O, cryst. from Me_2CO. M.p. 128°. Very sol. H_2O. Sol. EtOH, hot Me_2CO. $[\alpha]_D^{20} + 22{\cdot}2°$ in H_2O.

Di-Me ester: $C_7H_{12}O_7$. MW, 208. *Tri-Me ether*: $C_{10}H_{18}O_7$. MW, 250. B.p. about 143°/15 mm., 100°/0·1 mm. n_D^{21} 1·4353. $[\alpha]_D^{20} - 47{\cdot}5°$ in MeOH, $[\alpha]_D^{20} - 42{\cdot}5°$ in H_2O.

Diamide: $C_5H_{10}O_5N_2$. MW, 178. *Tri-Me ether*: $C_8H_{16}O_5N_2$. MW, 220. M.p. 232–3° decomp. $[\alpha]_D^{20} - 49°$ in H_2O.

l-.

Plates from EtOH. M.p. 127°. $[\alpha]_D^{18} - 23{\cdot}3°$ in H_2O. $k = 1{\cdot}32 \times 10^{-3}$ at 25°. *Ag salt*: m.p. 173°. *Quinine salt*: needles + $5H_2O$ from H_2O. M.p. 180°. $[\alpha]_D - 112{\cdot}5°$. *Brucine salt*: needles from EtOH.Aq. M.p. 175–6°. $[\alpha]_D - 41{\cdot}67°$.

Di-Me ester: *tri-Me ether*, syrup. B.p. 143°/18 mm., 74–6°/·005 mm. Sol. H_2O and most org. solvents. n_D^{20} 1·4350. $[\alpha]_D + 47{\cdot}3°$ in MeOH, $[\alpha]_D + 45°$ in H_2O.

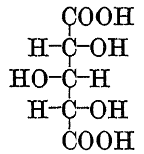
Diamide: *tri-Me ether*, prisms from MeOH. M.p. 232–3°. $[\alpha]_D + 50{\cdot}0°$ in H_2O.

dl-.

Cryst. from Me_2CO. M.p. 154·5° decomp. Very sol. H_2O, EtOH. Sol. Me_2CO. $k = 6{\cdot}9 \times 10^{-4}$ at 25°.

Hirst, Smith, *J. Chem. Soc.*, 1928, 3153.
Votoček, *Ber.*, 1910, **43**, 472.
Nef, *Ann.*, 1914, **403**, 252.
Ruff, *Ber.*, 1899, **32**, 558.
Haworth, Hirst, Jones, *J. Chem. Soc.*, 1927, 2428.

II. Xylo-trihydroxyglutaric Acid.



Cryst. from AcOEt. M.p. 152° decomp. Very sol. H_2O, hot EtOH. Spar. sol. hot Me_2CO. Insol. Et_2O, $CHCl_3$. Optically inactive. Heat of comb. C_v 389·5 Cal. $k = 6{\cdot}6 \times 10^{-4}$ at 25°. Evap. aq. sol. ⟶ lactone. HI(+ P) ⟶ glutaric acid. Reduces $NH_3.AgNO_3$ but not Fehling's.

Di-phenylhydrazide: plates. Sinters at 175°, decomp. at 210°.

Di-Me ester: 1 : 2-*Di-Me ether*. $C_9H_{16}O_7$. MW, 236. Oil. B.p. 132°/12 mm. 97–9°/0·003 mm. Hyd. ⟶ lactone of 1 : 2-dimethoxyglutaric acid.

Diamide: *tri-Me ether*, m.p. 195–8° (194–5°).

Schmidt, Zeiser, *Ber.*, 1934, **67**, 2124.
Fischer, Herborn, *Ber.*, 1896, **29**, 1965.
Haworth, Jones, *J. Chem. Soc.*, 1927, 2349.
Hirst, *J. Chem. Soc.*, 1926, 350.

Trihydroxyheptane.
See Heptantriol.

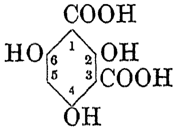
Trihydroxyhexane.
See Hexantriol.

1 : 6 : 8-Trihydroxy-3-hydroxymethyl-anthraquinone.
Me ether : *see* Carviolin.

2 : 4 : 6-Trihydroxyisobutyrophenone.
See Phlorisobutyrophenone.

5 : 7 : 4′-Trihydroxyisoflavone.
See Genistein.

2 : 4 : 6-Trihydroxyisophthalic Acid (*Phloroglucinol-dicarboxylic acid*)



$C_8H_6O_7$ MW, 214

Free acid unknown.
Di-Me ester : $C_{10}H_{10}O_7$. MW, 242. Needles or prisms from MeOH. M.p. 145–6°.
Di-Et ester : $C_{12}H_{14}O_7$. MW, 270. Needles from EtOH. M.p. 104° (107–8°). *Triacetyl* : prisms from EtOH. M.p. 96–8°.
Tri-Me ether : $C_{11}H_{12}O_7$. MW, 256. Prisms from H_2O. Sinters at 260°. *Di-Me ester* : $C_{13}H_{16}O_7$. MW, 284. Plates from Et_2O. M.p. 120–1°. Sol. $CHCl_3$, Me_2CO, AcOEt, AcOH, C_6H_6. Mod. sol. MeOH, Et_2O. Spar. sol. ligroin. *Di-Et ester* : $C_{15}H_{20}O_7$. MW, 312. Plates from EtOH. M.p. 90–1°. Sol. EtOH, Et_2O. Spar. sol. pet. ether.

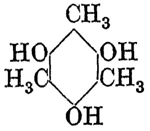
Leuchs, *Ann.*, 1928, **460**, 1.
Leuchs, Dzieng, *Ann.*, 1924, **440**, 151.
Leuchs, Simion, *Ber.*, 1911, **44**, 1878.

4 : 5 : 6-Trihydroxyisophthalic Acid (*Gallocarboxylic acid, pyrogallol-*4 : 6*-dicarboxylic acid*).

Needles + $3H_2O$ from H_2O. M.p. 283° decomp. Hot Ac_2O ⟶ pyrogallol triacetate.
Tri-Me ether : prisms from MeOH. M.p. 191°. *Di-Me ester* : plates from MeOH. M.p. 35–6°.

Feist, Awe, *Ber.*, 1926, **59**, 175.

2 : 4 : 6-Trihydroxymesitylene (2 : 4 : 6-*Trimethylphloroglucinol*)



$C_9H_{12}O_3$ MW, 168

Needles from AcOH. M.p. 184°. Sol. MeOH, EtOH, AcOEt, hot H_2O. Spar. sol. C_6H_6, hot pet. ether. Reduces $NH_3.AgNO_3$. $FeCl_3$ ⟶ reddish-violet col.
Mono-Me ether : $C_{10}H_{14}O_3$. MW, 182. Needles from H_2O or C_6H_6. M.p. 121°. B.p. 196–8°/20 mm. *Diacetyl* : cryst. from MeOH.Aq. M.p. 66–8°.
Mono-Et ether : $C_{11}H_{16}O_3$. MW, 196. Cryst. from $CHCl_3$. M.p. 130°.
Triacetyl : prisms from C_6H_6. M.p. 165–7°.

Weidel, Wenzel, *Monatsh.*, 1898, **19**, 257

Trihydroxymethane.
See Orthoformic Acid.

5 : 7 : 3′-Trihydroxy-4′-methoxyflavanone.
See Hesperetin.

5 : 7 : 4′-Trihydroxy-3′-methoxyflavanone.
See Homoeriodictyol.

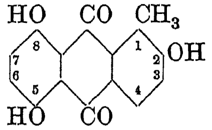
3 : 5 : 7-Trihydroxy-4′-methoxyflavone.
See Kaempferide.

5 : 7 : 3′-Trihydroxy-4′-methoxyflavone.
See Diosmetin.

5 : 7 : 4′-Trihydroxy-3′-methoxyflavone.
See Chrysoeriol.

5 : 7 : 4′ - Trihydroxy - 6 - methoxyiso flavone.
See Tectorigenin.

2 : 5 : 8 - Trihydroxy - 1 - methylanthra - quinone



$C_{15}H_{10}O_5$ MW, 270

Dark red needles from $CHCl_3$. M.p. 270°. Sol. alkalis and conc. H_2SO_4 with bluish-red col.
2-*Me ether* : $C_{16}H_{12}O_5$. MW, 284. Dark red needles from AcOH. M.p. 249–249·5°. Sol. conc. H_2SO_4 with bluish-violet col., in alkalis with bluish-red col.

Gardner, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 2455.
Graves, Adams, *ibid.*, 2439.

3 : 5 : 8 - Trihydroxy - 1 - methylanthra - quinone.
Red needles from EtOH. M.p. 260°.
Triacetyl : yellow needles from C_6H_6. M.p. 179°.

Dimroth, Fick, *Ann.*, 1916, **411**, 330.

3 : 7 : 8 - Trihydroxy - 1 - methylanthra - quinone.
See 8-Methylflavopurpurin.

4 : 5 : 8 - Trihydroxy - 1 - methylanthra - quinone.
Red needles from AcOH. M.p. 276–8°. Mod. sol. cold EtOH. Sublimes. Sol. alkalis with violet-red col., in conc. H_2SO_4 with blue to bluish-violet col. Alc. $FeCl_3$ ⟶ greenish-brown col.
5 : 8-*Di-Me ether* : $C_{17}H_{14}O_5$. MW, 298. Red needles from Me_2CO. M.p. 224°. Sol. alkalis with red col., in conc. H_2SO_4 with blue to bluish-red col.

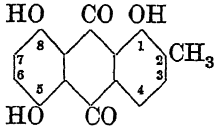
Tri-Me ether: $C_{18}H_{16}O_5$. MW, 312. Yellow needles from EtOH. M.p. 249–250·5°.
Triacetyl: m.p. 197°.

Keimatsu, Hirano, Yoshimi, *Chem. Zentr.*, 1930, II, 2384.
Gardner, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 2455.
Graves, Adams, *ibid.*, 2439.

4 : 7 : 8 - Trihydroxy - 1 - methylanthra - quinone.
Red needles from AcOH. M.p. 301° (decomp. at 290°).
7 : 8-*Di-Me ether*: yellow needles from AcOEt. M.p. 168–9°. *Acetyl*: yellow needles from EtOH. M.p. 173–4°.
Triacetyl: yellow needles from EtOH. M.p. 204–5°.

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2011.

5 : 6 : 7 - Trihydroxy - 1 - methylanthra - quinone.
See 5-Methylanthragallol.
6 : 7 : 8 - Trihydroxy - 1 - methylanthra - quinone.
See 8-Methylanthragallol.
1 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.
See Morindone.
1 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone



$C_{15}H_{10}O_5$ MW, 270
Red needles from AcOH. M.p. 253–4°. Very sol. Et_2O, C_6H_6, toluene. Mod. sol. EtOH. Sublimes at 250–60°. Sol alkalis with red col., in conc. H_2SO_4 with bluish-red col. Alc. $FeCl_3 \longrightarrow$ red col.
5 : 8-*Di-Me ether*: $C_{17}H_{14}O_5$. MW, 298. Red needles from AcOH. M.p. 165°. Sol. alkalis with red col., in conc. H_2SO_4 with blue to bluish-red col.
Tri-Me ether: $C_{18}H_{16}O_5$. MW, 312. Red needles from EtOH. M.p. 206·5–207°.
Triacetyl: yellow needles. M.p. 215°.

Keimatsu, Hirano, *Chem. Zentr.*, 1930, II, 1551.
Graves, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 2439.

1 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.
Brownish-yellow needles from AcOH.Aq. M.p. 287–8°. Sol. NaOH with red col., in conc. H_2SO_4 with bluish-red col.
Tri-Me ether: yellow needles from AcOEt. M.p. 209–10°. Sol. conc. H_2SO_4 with reddish-purple col.

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 288.
Simonsen, *J. Chem. Soc.*, 1924, **125**, 726.

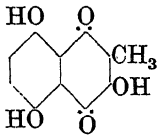
3 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.
Orange-red needles from $PhNO_2$. Does not melt below 330°.
5 : 6-*Di-Me ether*: $C_{17}H_{14}O_5$. MW, 298. Yellow needles from AcOH. M.p. about 310°.
Triacetyl: yellow needles from EtOH. M.p. 232–3°.

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2011.

3 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone.
See Rhababerone.
3 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.
See 7-Methylflavopurpurin.
4 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.
Tri-Me ether: needles from AcOH.Aq. M.p. 164–5°. Sol. conc. H_2SO_4 with reddish-purple col.

Simonsen, *J. Chem. Soc.*, 1924, **125**, 724.

4 : 5 : 7 - Trihydroxy - 2 - methylanthra - quinone.
See Frangula-emodin.
4 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone.
See Helminthosporin.
5 : 6 : 7 - Trihydroxy - 2 - methylanthra - quinone.
See 6-Methylanthragallol.
6 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.
See 7-Methylanthragallol.
5 : 7 : 4′-Trihydroxy-2-methylisoflavone.
See 2-Methylgenistein.
3 : 5 : 8-Trihydroxy-2-methyl-1 : 4-naphthoquinone (*Hydroxydroserone*)

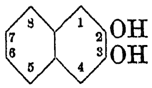


$C_{11}H_8O_5$ MW, 220
Occurs in insectiverous plant *Drosera Whittakeri*. Red plates from AcOH. M.p. 192–3°.
3-*Acetyl*: cryst. from C_6H_6. M.p. 151–6°.
Triacetyl: m.p. 152–3°.

Lugg, Macbeth, Winzor, *J. Chem. Soc.*, 1936, 1457.
Kuroda, *Proc. Imper. Acad., Tokyo*, 1939, **15**, 226, (*Chem. Abstracts*, 1939, **33**, 8599).

1 : 4 : 5-Trihydroxy-2-methylxanthone.
See Ravenelin.

1 : 2 : 3-Trihydroxynaphthalene (*Naphthopyrogallol*)



$C_{10}H_8O_3$ MW, 176

Prisms. Decomp. above 250°. Sol. H_2O, EtOH, Me_2CO.

Triacetyl : prisms from AcOH. M.p. 250–5°.

Zincke, Noack, *Ann.*, 1897, **295**, 17.

1 : 2 : 4-Trihydroxynaphthalene.

Needles from C_6H_6. M.p. 154°. Sol. H_2O, EtOH, Et_2O, $CHCl_3$. Spar. sol. C_6H_6.

Triacetyl : plates from EtOH.Aq. M.p. 134·5°.

Bayer, D.R.P., 101,607, (*Chem. Zentr.*, 1899, I, 1094).
Thiele, Winter, *Ann.*, 1911, **311**, 345.

1 : 2 : 7-Trihydroxynaphthalene.

Cryst. from ligroin or xylene. M.p. 197°.

7-*Me ether* : leaflets from ligroin. M.p. 127°. *Diacetyl* : needles from EtOH.Aq. M.p. 122°. *Picrate* : needles from EtOH. M.p. 156°.

Tri-Me ether : picrate; red needles from EtOH.Aq. M.p. 115°.

Triacetyl : cryst. from toluene. M.p. 181–2°.

Chakravarti, Pasupati, *J. Chem. Soc.*, 1937, 1859.

1 : 3 : 6-Trihydroxynaphthalene.

Cryst. from H_2O. M.p. 95°. Sol. H_2O, EtOH, Et_2O, Me_2CO. Spar. sol. $CHCl_3$, C_6H_6, pet. ether. $FeCl_3$ ⟶ yellowish-brown col.

Triacetyl : needles from EtOH.Aq. M.p. 112–13°.

Meyer, Hartmann, *Ber.*, 1905, **38**, 3950.

1 : 4 : 5-Trihydroxynaphthalene.
See α-Hydrojuglone.

1 : 4 : 6-Trihydroxynaphthalene.

Needles from Et_2O–pet. ether. M.p. 138–40°. Very unstable in moist air.

Triacetyl : needles from EtOH. M.p. 94–5°.

Fischer, Bauer, *J. prakt. Chem.*, 1916, **94**, 8.

1 : 6 : 7-Trihydroxynaphthalene.

Yellow needles from xylene. M.p. 177°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6. $FeCl_3$ ⟶ blue col.

Tri-Me ether : $C_{13}H_{14}O_3$. MW, 218. Prisms from ligroin. M.p. 127–8°.

Triacetyl : greenish cryst. from EtOH.Aq. M.p. 143–4°.

Friedländer, Silberstern, *Monatsh.*, 1902, **23**, 530.

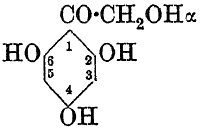
6 : 7 : 8-Trihydroxy-1-naphthoic Acid.
See Purpurogallone.

5 : 6 : 8-Trihydroxy-1 : 4-naphthoquinone.
See Naphthopurpurin.

8 : 9 : 15-Trihydroxypalmitic Acid.
See Aleuritic Acid.

Trihydroxypentane.
See Pentantriol.

2 : 4 : 6 - Trihydroxyphenacyl Alcohol (2 : 4 : 6 : α-*Tetrahydroxyacetophenone*)



$C_8H_8O_5$ MW, 184

α-*Me ether* : $C_9H_{10}O_5$. MW, 198. Needles from hot H_2O. M.p. 192°. $FeCl_3$ ⟶ violet col.

4 : 6 : α-*Tri-Me ether* : $C_{11}H_{14}O_5$. MW, 226. Needles from EtOH. M.p. 102–4°. *Oxime* : needles from MeOH.Aq. M.p. 147–9°.

Tetra-Me ether : $C_{12}H_{16}O_5$. MW, 240. Cryst. from MeOH. M.p. 50°. No col. with $FeCl_3$.

4 : 6 : α-*Tri-Et ether* : $C_{14}H_{20}O_5$. MW, 268. Needles from EtOH. M.p. 96–7°.

Herzig, Hoffmann, *Ber.*, 1909, **42**, 156.
Perkin, *J. Chem. Soc.*, 1911, **99**, 1724.
Slater, Stephen, *J. Chem. Soc.*, 1920, **117**, 316.

3 : 4 : 5 - Trihydroxyphenacyl Alcohol (3 : 4 : 5 : α-*Tetrahydroxyacetophenone*).

α-*Me ether* : *triacetyl*, cryst. from EtOH. M.p. 132–3°.

3 : 5-*Di-Me ether* : $C_{10}H_{12}O_5$. MW, 212. Needles + H_2O from boiling H_2O. M.p. 93–5°, anhyd. 132°. Reduces cold Fehling's. Alc. $FeCl_3$ ⟶ weak olive-green col. *Diacetyl* : prismatic needles from MeOH. M.p. 123°. 4-*Benzoyl* : prismatic needles from 50% EtOH. M.p. 173–5°. *Dibenzoyl* : micro-needles from C_6H_6–pet. ether. M.p. 128°.

3 : 4 : 5-*Tri-Me ether* : α-*acetyl*, m.p. 88°.

Tetra-Me ether : needles from C_6H_6–pet. ether. M.p. 54°. B.p. 212°/15 mm. *Semicarbazone* : needles from EtOH.Aq. M.p. 158°.

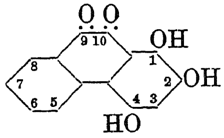
3 : 4 : 5-*Triacetyl* : needles from C_6H_6. M.p. 87–8°. Sol. Et_2O, Me_2CO, AcOEt, C_6HCl_3, hot EtOH, C_6H_6. Spar. sol. H_2O. Insol. pet. ether. No col. with $FeCl_3$. Reduces cold Fehling's.

Tetra-acetyl : cryst. from EtOH. M.p. 124–5°.

3 : 4 : 5-*Tribenzoyl* : α-*formyl*, prismatic needles from formic acid. M.p. 116–17°. α-*Acetyl* : pale yellow plates from EtOH. M.p. 138–40°. α-p-*Toluenesulphonyl* : needles. M.p. 148°.

Pratt, Robinson, *J. Chem. Soc.*, 1925, 173.
Levy, Posternack, Robinson, *J. Chem. Soc.*, 1931, 2705.
Reynolds, Robinson, *J. Chem. Soc.*, 1934, 1040.

1 : 2 : 4-Trihydroxyphenanthraquinone



$C_{14}H_8O_5$ MW, 256

Very readily oxidised in air. Very sol. EtOH. Spar. sol. most other org. solvents. Does not crystallise, and decomposes on heating. Sol. alkalis with green col. ⟶ pale red in air. Sol. conc. H_2SO_4 with green col., in Py with cornflower-blue col.

Triacetyl: orange plates from toluene. M.p. 227–8° decomp.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 1939.

1 : 3 : 4-Trihydroxyphenanthraquinone. Dark red cryst. from EtOH. Very unstable to air. Spar. sol. H_2O. Sol. alkalis with green col. ⟶ pink in air. Sol. conc. H_2SO_4 with red col., in Py with red col. ⟶ green on dilution with H_2O.

Triacetyl: yellow needles from AcOH. Decomp. at 240°.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 1940.

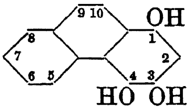
2 : 3 : 4-Trihydroxyphenanthraquinone. Brownish-red powder. M.p. 185° decomp.

Monosemicarbazone: brownish-red powder from EtOH. Decomp. at 270°.

Phenazine: $C_{20}H_{12}O_3N_2$. Brown microcryst. from EtOH. M.p. 255° decomp.

Schmidt, Schairer, *Ber.*, 1923, **56**, 1337.

1 : 3 : 4-Trihydroxyphenanthrene



$C_{14}H_{10}O_3$ MW, 226

Triacetyl: needles from C_6H_6–pet. ether. M.p. 138°.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 1940.

1 : 5 : 6-Trihydroxyphenanthrene.

1 : 5-*Di-Me ether*: $C_{16}H_{14}O_3$. MW, 254. Reddish-brown plates from EtOH.Aq. M.p. 164–5°. *Acetyl*: prisms from AcOH. M.p. 96–7°.

5 : 6-*Di-Me ether*: prisms from EtOH. M.p. 182–3°. 1-*Et ether*: $C_{18}H_{18}O_3$. MW, 282. Plates from MeOH. M.p. 100°.

Tri-Me ether: $C_{17}H_{16}O_3$. MW, 268. Plates from MeOH. M.p. 138°.

Pschorr, *Ber.*, 1912, **45**, 2220; 1900, **33**, 181.

3 : 4 : 5-Trihydroxyphenanthrene. Plates from H_2O. M.p. 148°. Sol. EtOH, Et_2O, $CHCl_3$.

Tri-Me ether: cryst. from MeOH. M.p. 90°. *Picrate*: red plates from MeOH. M.p. 166°.

Pschorr, *Ann.*, 1912, **391**, 53.

3 : 4 : 6-Trihydroxyphenanthrene.

3 : 6-*Di-Me ether*: *see* Thebaol.

Tri-Me ether: oil. *Picrate*: red needles from EtOH. M.p. 109–10° (110–12°).

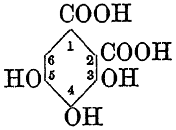
Pschorr, Seydel, Stöhrer, *Ber.*, 1902, **35**, 4406.

Vongerichten, *Ber.*, 1902, **35**, 4411.

Trihydroxyphenyl hydroxystyryl Ketone.

See Tetrahydroxychalkone.

3 : 4 : 5-Trihydroxyphthalic Acid (*Pyrogallol-4 : 5-dicarboxylic acid*)



$C_8H_6O_7$ MW, 214

3 : 5-*Di-Me ether*: $C_{10}H_{10}O_7$. MW, 242. Cryst. from H_2O. M.p. 225–7°. *Anhydride*: $C_{10}H_8O_6$. MW, 224. Needles from toluene. M.p. 177–9°.

Tri-Me ether: $C_{11}H_{12}O_7$. MW, 256. Plates from H_2O. M.p. 176–7° decomp. $FeCl_3$ ⟶ yellow col. 1-*Me ester*: $C_{12}H_{14}O_7$. MW, 270. Needles from MeOH. M.p. 138–41°. *Di-Me ester*: $C_{13}H_{16}O_7$. MW, 284. Cryst. M.p. 64–5°. *Anhydride*: $C_{11}H_{10}O_6$. MW, 238. Needles from C_6H_6. M.p. 147°.

Alimchandani, Meldrum, *J. Chem. Soc.*, 1920, **117**, 964.

Feist, Dschu, *Chem. Zentr.*, 1927, II, 58.

3 : 4 : 6-Trihydroxyphthalic Acid.

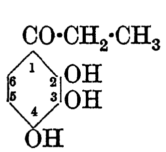
Tri-Me ether: prisms + $1H_2O$ from H_2O. M.p. 216–17° (185°).

Faltis, Kloiber, Gutlohn, Attia, *Monatsh.*, 1929, **53** and **54**, 632.

Trihydroxypropane.

See Glycerol *and* Orthopropionic Acid.

2 : 3 : 4-Trihydroxypropiophenone (4-*Propionylpyrogallol*)



$C_9H_{10}O_4$ MW, 182

Yellow needles. M.p. 127°.

Badische, D.R.P., 42,149.

2 : 4 : 5-Trihydroxypropiophenone.

4 : 5-*Di-Me ether*: $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH.Aq. M.p. 124–6°. $FeCl_3$ ⟶ green col. *Acetyl*: needles from EtOH.Aq. M.p. 117–18°. *Benzoyl*: needles. M.p. 110–11°.

Tri-Me ether: $C_{12}H_{16}O_4$. MW, 224. Needles from H_2O. M.p. 106–8°. *Oxime*: plates from EtOH.Aq. M.p. 106–8°. *Semicarbazone*: (i) plates from EtOH.Aq. M.p. 182. (ii) Prisms from H_2O. M.p. 166–7°.

Bargellini, Martegiani, *Gazz. chim. ital.*, 1911, **41**, 449.

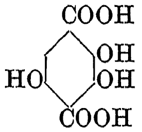
2 : 4 : 6-Trihydroxypropiophenone.
See Phlorpropiophenone.

3 : 4 : 5-Trihydroxypropiophenone.
Tri-Me ether: needles from ligroin. M.p. 51–2°. Sol. EtOH, AcOH, warm ligroin. p-*Nitrophenylhydrazone*: red needles from EtOH. M.p. 182–3°.

Mauthner, *J. prakt. Chem.*, 1926, **112**, 269.

3 : 5 : 4′-Trihydroxystilbene.
See Resveratrol.

2 : 3 : 5-Trihydroxyterephthalic Acid

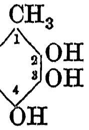


$C_8H_6O_7$ MW, 214

Orange-yellow prisms from AcOH. M.p. 247° decomp. Sol. common org. solvents.
Di-Et ester: $C_{12}H_{14}O_7$. MW, 270. Yellow needles from Me_2CO. M.p. 116°. *Triacetyl*: cryst. from Me_2CO.Aq. M.p. 100°.

Liebermann, Lisser, *Ann.*, 1934, **513**, 184.

2 : 3 : 4-Trihydroxytoluene (4-*Methylpyrogallol*)



$C_7H_8O_3$ MW, 140

Needles. M.p. 140–1°.

Majima, Okazaki, *Ber.*, 1916, **49**, 1492.

2 : 3 : 5-Trihydroxytoluene.
3-*Me ether*: $C_8H_{10}O_3$. MW, 154. Needles. M.p. 128–9°. Sol. EtOH, Et_2O, AcOH. Spar. sol. ligroin. Reduces Fehling's.
2 : 3-*Di-Me ether*: $C_9H_{12}O_3$. MW, 168. Plates from C_6H_6. M.p. 140–1°.

Henrich, Nachtigall, *Ber.*, 1903, **36**, 894.

2 : 3 : 6-Trihydroxytoluene.
2-*Me ether*: needles from ligroin. M.p. 117–18°.

Majima, Okazaki, *Ber.*, 1916, **49**, 1490.

2 : 4 : 5-Trihydroxytoluene.
Prisms from C_6H_6. M.p. 131–2°. Sol. H_2O. Aq. sol. oxidises readily.
4-*Me ether*: $C_8H_{10}O_3$. MW, 154. Needles from H_2O. M.p. 124°. Sol. common org. solvents, hot H_2O.
4-*Et ether*: $C_9H_{12}O_3$. MW, 168. Tablets from C_6H_6. M.p. 131°. Sublimes.
Tri-Me ether: $C_{10}H_{16}O_3$. MW, 182. Plates from MeOH.Aq. M.p. 55°. Sol. Et_2O, EtOH, pet. ether.
Triacetyl: cryst. from EtOH. M.p. 114–15°.

Thiele, Winter, *Ann.*, 1900, **311**, 349.
Luff, Perkin, Robinson, *J. Chem. Soc.*, 1910, **97**, 1137.
Bayer, D.R.P., 101,607, (*Chem. Zentr.*, 1899, I, 1094).

2 : 4 : 6 - Trihydroxytoluene (2-*Methylphloroglucinol*).
Needles from AcOEt. M.p. 214–16°. Sol. H_2O, EtOH, Et_2O, AcOEt, hot AcOH. Insol. C_6H_6, pet. ether. Sublimes.
2-*Me ether*: $C_8H_{10}O_3$. MW, 154. Prisms + $1H_2O$ from H_2O. M.p. 91°, anhyd. 117–19°. Sol. EtOH, Et_2O, hot H_2O, hot C_6H_6.
4-*Me ether*: needles from xylene. M.p. 124°. B.p. 195–8°/20 mm. Sol. EtOH, AcOEt. Spar. sol. H_2O, ligroin.
2 : 4-*Di-Me ether*: $C_9H_{12}O_3$. MW, 168. Needles from xylene–ligroin. M.p. 60–1°. B.p. 178–80°/20 mm. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O, ligroin. 6-*Et ether*: $C_{11}H_{16}O_3$. MW, 196. Cryst. from 75% EtOH. M.p. 38°. B.p. 149–51°/16 mm.
2 : 4 : 6-*Tri-Me ether*: $C_{10}H_{14}O_3$. MW, 182. Cryst. M.p. 10–13°. B.p. 140–2°/18 mm.
4-*Et ether*: $C_9H_{12}O_3$. MW, 168. Needles from C_6H_6. M.p. 136–7°. B.p. 195–200°/13 mm. Sol. EtOH, Et_2O. *Diacetyl*: plates from EtOH. M.p. 91°.
Triacetyl: needles from ligroin. M.p. 76° (52°).

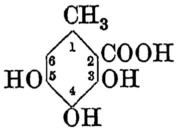
Herzig, Wenzel, *Monatsh.*, 1916, **37**, 573.
Boehm, *Ann.*, 1901, **318**, 286.
Herzig, Theuer, *Monatsh.*, 1900, **21**, 855.

3 : 4 : 5-Trihydroxytoluene (5-*Methylpyrogallol*).
Needles from C_6H_6. M.p. 120°. Sublimes in needles.
3 : 5-*Di-Me ether*: cryst. from EtOH. M.p. 36°. B.p. 265°, 145–6°/12 mm. Sol. H_2O, Et_2O.
3 : 4-*Di-Me ether*: *see* Iridol.
Tri-Me ether: *see under* Iridol.
Triacetyl: cryst. M.p. 99°.

Rosauer, *Monatsh.*, 1898, **19**, 565.
Hofmann, *Ber.*, 1879, **12**, 1376.

ω-Trihydroxytoluene.
See Orthobenzoic Acid.

3 : 4 : 5-Trihydroxy-o-toluic Acid



$C_8H_8O_5$ MW, 184

Me ester: $C_9H_{10}O_5$. MW, 198. Cryst. M.p. 155°.

Koller, Hamburg, *Monatsh.*, 1935, **65**, 373.

2 : 4 : 6-Trihydroxy-*m*-toluic Acid (4-*Methylphloroglucinol-2-carboxylic acid*).

M.p. 177°.

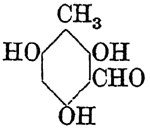
4-*Me ether*: $C_9H_{10}O_5$. MW, 198. Cryst. from Et_2O–C_6H_6. M.p. 147° decomp. Sol. EtOH. Insol. H_2O, ligroin. *Me ester*: $C_{10}H_{12}O_5$. MW, 212. Cryst. from EtOH. M.p. 132–3°. Spar. sol. EtOH. *Diacetyl*: cryst. from EtOH. M.p. 75–7°.

Me ester: cryst. from MeOH. M.p. 144–5°.

Triacetyl: cryst. from C_6H_6. M.p. 163–4°.

Schreier, Wenzel, *Monatsh.*, 1904, **25**, 312.
Herzig, Wenzel, Graetz, *Monatsh.*, 1902, **23**, 100.

2 : 4 : 6-Trihydroxy-*m*-toluic Aldehyde



$C_8H_8O_4$ MW, 168

Cryst. + $\frac{1}{2}H_2O$ from H_2O. Decomp. at 130°. Sol. EtOH, AcOH, AcOEt. Spar. sol. H_2O, Et_2O. Insol. $CHCl_3$, C_6H_6, ligroin.

Oxime: yellow cryst. Decomp. at 140°.

Herzig, Wenzel, Kerényi, *Monatsh.*, 1903, **24**, 876.

2 : 3 : 5-Trihydroxy-*p*-toluic Aldehyde.

See Thamnol.

2 : 2′ : 2″-Trihydroxytriethylamine (*Triethanolamine*)

$N(CH_2{\cdot}CH_2OH)_3$

$C_6H_{15}O_3N$ MW, 149

Oil. B.p. 277–9°/150 mm. D^{20} 1·1242. n_D^{20} 1·4852. Darkens in air. Non-volatile in steam. Completely miscible with H_2O, EtOH. Sol. $CHCl_3$. Spar. sol. Et_2O, C_6H_6, ligroin.

B,HCl: cryst. from EtOH. M.p. 177°. Spar. sol. H_2O, EtOH.

B,HAuCl$_4$: plates + H_2O. M.p. anhyd. 77–8°.

B$_2$,H$_2$PtCl$_6$: cryst. from EtOH. M.p. 118–19°.

Triacetyl: oil. B.p. 206–7°/27 mm. Very sol. EtOH, Et_2O. Insol. H_2O.

N-*Oxide*: cryst. from EtOH. M.p. 104–105·5°. Very sol. H_2O, EtOH. Spar. sol. $CHCl_3$, Me_2CO. Insol. AcOEt, ligroin. Reduces hot NH_3.$AgNO_3$ but not Fehling's. Sweet taste. *Picrate*: deliquescent cryst. from EtOH–Et_2O. M.p. 73–4°.

I.G., F.P., 650,574, (*Chem. Abstracts*, 1929, **23**, 3232).
Jones, Burns, *J. Am. Chem. Soc.*, 1925, **47**, 2966.
Wurtz, *Ann.*, 1862, **121**, 227.

5 : 7 : 5′-Trihydroxy-6 : 3′ : 4′-trimethoxyisoflavone.

See Irigenin.

4 : 6 : 3′-Trihydroxy-5 : 2 : 5′-trimethyl-2-hydroxymethyl-diphenyl Ether.

See Hyposalazinol.

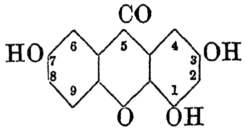
4 : 4′ : 4″-Trihydroxytriphenylmethane.

See Leucaurine.

3 : 6 : 7-Trihydroxytropane.

See Teloidine.

1 : 3 : 7-Trihydroxyxanthone



$C_{13}H_8O_5$ MW, 244

Yellow needles. M.p. 314–15°. NaOH.Aq. ⟶ orange salt and yellow sol. $FeCl_3$ ⟶ brownish-green col.

Triacetyl: m.p. 225–6°.

Anand, Venkataraman, *Chem. Abstracts*, 1948, **42**, 6810.

1 : 4 : 7-Trihydroxyxanthone.

Cryst. from AcOEt–C_6H_6. M.p. 300–2°.

Triacetyl: cryst. from EtOH. M.p. 188–90°.

Rao, Seshadri, *Chem. Abstracts*, 1948, **42**, 3394.

2 : 3 : 6-Trihydroxyxanthone.

Pale yellow needles + $2H_2O$ from EtOH.Aq. M.p. 328–30° decomp. Sol. EtOH. Spar. sol. H_2O. Insol. Me_2CO, Py. Sol. alkalis with yellow col.

6-*Me ether*: $C_{14}H_{10}O_5$. MW, 258. Needles from EtOH. Decomp. at 249–51°. *Diacetyl*: needles from EtOH. M.p. 231–2° decomp.

2 : 3-*Di-Me ether*: $C_{15}H_{12}O_5$. MW, 272. Yellow needles from EtOH. M.p. 228–31° decomp. *Acetyl*: needles from EtOH. M.p. 224–5° decomp.

Tri-Me ether: $C_{16}H_{14}O_5$. MW, 286. Yellow needles from EtOH. M.p. 194–5°.

Triacetyl: yellow needles from EtOH. M.p. 226–7°.

Nierenstein, *Ber.*, 1913, **46**, 650.
Dean, Nierenstein, *J. Chem. Soc.*, 1920, 802.

2 : 4 : 7-Trihydroxyxanthone.

See Gentisein.

Tri-indole.

See under Indole.

Tri-iodoacetanilide.

See under Tri-iodoaniline.

Tri-iodoacetic Acid

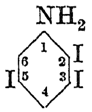
$CI_3{\cdot}COOH$

$C_2HO_2I_3$ MW, 438

Yellow leaflets. M.p. 150° decomp. Warm AcOH ⟶ CO_2 + iodoform.

Angeli, *Ber.*, 1893, **26**, 596.

2 : 3 : 5-Tri-iodoaniline



$C_6H_4NI_3$ MW, 471

Needles. M.p. 116°. Mod. sol. EtOH, AcOH, C_6H_6. Spar. sol. Et_2O, ligroin.

Acetyl : 2 : 3 : 5-tri-iodoacetanilide. Needles. M.p. 227°. Sublimes slowly above 200°. Spar. sol. usual solvents.

Brenans, *Bull. soc. chim.*, 1904, **31**, 131.

2 : 3 : 6-Tri-iodoaniline.

Needles from EtOH or EtOH–Et_2O. M.p. 116·8°.

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 689.

2 : 4 : 5-Tri-iodoaniline.

Needles from EtOH–Et_2O. M.p. 117·8°.

Acetyl : 2 : 4 : 5-tri-iodoacetanilide. Needles. M.p. 241·5°. Spar. sol. EtOH, Et_2O.

Körner, Belario, *Atti accad. Lincei*, 1908, **17**, 1, 689.

2 : 4 : 6-Tri-iodoaniline.

Needles from EtOH. M.p. 185·5° (184°). Sol. AcOH, CS_2. Mod. sol. hot EtOH.

Wheeler, Johns, *Am. Chem. J.*, 1910, **43**, 405.

Michael, Norton, *Ber.*, 1878, **11**, 111.

Jackson, Whitmore, *J. Am. Chem. Soc.*, 1915, **37**, 1528.

3 : 4 : 5-Tri-iodoaniline.

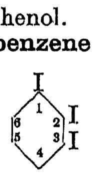
Needles from EtOH–Me_2CO. M.p. 174·5° decomp. Very sol. $CHCl_3$, Me_2CO, pet. ether. Sol. C_6H_6. Mod. sol. EtOH, Et_2O.

Kalb, Schweizer, Zellner, Berthold, *Ber.*, 1926, **59**, 1867.

Tri-iodoanisole.

See under Tri-iodophenol.

1 : 2 : 3-Tri-iodobenzene



$C_6H_3I_3$ MW, 456

Needles from EtOH, prisms from C_6H_6. M.p. 116°. Very sol. EtOH, Et_2O, $CHCl_3$.

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 687.

See also Kalb, Schweizer, Zellner, Berthold, *Ber.*, 1926, **59**, 1862.

1 : 2 : 4-Tri-iodobenzene.

Needles from EtOH. M.p. 91·5° (77°). Sol. EtOH, $CHCl_3$.

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 683.

Brenans, *Bull. soc. chim.*, 1914, **15**, 383.

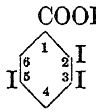
1 : 3 : 5-Tri-iodobenzene.

Needles from AcOH. M.p. 184·2° (180°). Sol. boiling AcOH. Spar. sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Insol. H_2O. Volatile in steam.

Jackson, Behr, *Am. Chem. J.*, 1901, **26**, 58.

Körner, Contardi, *Atti accad. Lincei*, 1913, **22**, I, 832.

2 : 3 : 5-Tri-iodobenzoic Acid



$C_7H_3O_2I_3$ MW, 500

Prisms from EtOH. M.p. 224–6° (223–4°). Sol. Et_2O, hot EtOH. Spar. sol. boiling C_6H_6. Insol. H_2O.

Wheeler, Johns, *Am. Chem. J.*, 1910, **43**, 407.

Klemme, Hunter, *J. Org. Chem.*, 1940, **5**, 509.

2 : 4 : 5-Tri-iodobenzoic Acid.

Needles from EtOH. M.p. 248°. Mod. sol. hot EtOH, Et_2O. Insol. hot H_2O, C_6H_6.

Wheeler, Johns, *Am. Chem. J.*, 1910, **44**, 451.

3 : 4 : 5-Tri-iodobenzoic Acid.

Prisms from EtOH. M.p. 288°. Sol. EtOH. Insol. H_2O.

Wheeler, Liddle, *Am. Chem. J.*, 1909, **42**, 458.

1 : 1 : 1-Tri-iodoethane (*Methyliodoform*)

$CH_3{\cdot}CI_3$

$C_2H_3I_3$ MW, 408

Yellow cryst. from EtOH. M.p. 95° decomp. Very sol. Et_2O, C_6H_6, CS_2. Spar. sol. EtOH, ligroin.

Boissieu, *Bull. soc. chim.*, 1888, **49**, 16.

Emschwiller, *Compt. rend.*, 1933, **196**, 1028.

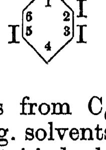
Tri-iodomethane.

See Iodoform.

Tri-iodophenetole.

See under Tri-iodophenol.

2 : 3 : 5-Tri-iodophenol



$C_6H_3OI_3$ MW, 472

Prismatic needles from C_6H_6–ligroin. M.p. 114°. Sol. usual org. solvents.

Et ether : 2 : 3 : 5-tri-iodophenetole. $C_8H_7OI_3$. MW, 500. Needles. M.p. 121°.

Acetyl : needles. M.p. 123°. Sol. EtOH, AcOH.

Brenans, *Bull. soc. chim.*, 1904, **31**, 132; 1914, **15**, 383.

2 : 4 : 6-Tri-iodophenol.

Needles from EtOH.Aq. M.p. 158–9°. Sol. 50 parts 95% EtOH. Mod. sol. Et_2O, Me_2CO. Spar. volatile in steam. Warm conc. $HNO_3 \longrightarrow$ picric acid.

Me ether: 2 : 4 : 6-tri-iodoanisole. $C_7H_5OI_3$. MW, 486. Leaflets from C_6H_6, needles from Et_2O. M.p. 98–9°.

Et ether: 2 : 4 : 6-tri-iodophenetole. Prismatic needles from Et_2O. M.p. 83°.

Propyl ether: $C_9H_9OI_3$. MW, 514. Needles. M.p. 81°.

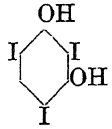
Allyl ether: $C_9H_7OI_3$. MW, 512. Needles from C_6H_6. M.p. 113–14°. Spar. sol. Et_2O.

Acetyl: needles or prisms from C_6H_6. M.p. 156°.

p-*Nitrobenzoyl*: m.p. 181°.

Brenans, *Bull. soc. chim.*, 1901, **25**, 630, 820.
Marsh, *J. Chem. Soc.*, 1927, 3164.
Brenans, Girod, *Compt. rend.*, 1928, **186**, 1851.

2 : 4 : 6-Tri-iodoresorcinol



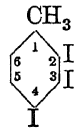
$C_6H_3O_2I_3$ MW, 488

Needles from C_6H_6. M.p. 149–50°. Sol. EtOH, Et_2O, CS_2. Spar. sol. hot H_2O.

Diacetyl: needles. M.p. 170°. Sol. EtOH, Et_2O.

Claassen, *Ber.*, 1878, **11**, 1442.
Michael, Norton, *Ber.*, 1876, **9**, 1752.
Rosenthaler, Capuano, *Chem. Abstracts*, 1948, **42**, 9077.

2 : 3 : 4-Tri-iodotoluene



$C_7H_5I_3$ MW, 470

Light brown needles from EtOH. M.p. 92°. Sol. C_6H_6. Mod. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 506.

2 : 3 : 5-Tri-iodotoluene.

Orange plates from EtOH. M.p. 72–3°. Spar. volatile in steam.

Wheeler, *Am. Chem. J.*, 1910, **44**, 506.

2 : 3 : 6-Tri-iodotoluene.

Needles from EtOH. M.p. 80·5°. Spar. sol. EtOH. Spar. volatile in steam.

Wheeler, *Am. Chem. J.*, 1910, **44**, 135.

2 : 4 : 5-Tri-iodotoluene.

Brownish needles from EtOH. M.p. 118–20°. Spar. volatile in steam.

Wheeler, *Am. Chem. J.*, 1910, **44**, 140, 500.
Cf. Neumann, *Ann.*, 1887, **241**, 56.

2 : 4 : 6-Tri-iodotoluene.

Needles from C_6H_6. M.p. 105°. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 501.
Cf. Neumann, *Ann.*, 1887, **241**, 56.

3 : 4 : 5-Tri-iodotoluene.

Needles from EtOH. M.p. 122–3°.

Wheeler, Liddle, *Am. Chem. J.*, 1909, **42**, 450.

Tri-isoamylamine

$[(CH_3)_2CH{\cdot}CH_2{\cdot}CH_2]_3N$

$C_{15}H_{33}N$ MW, 227

Liq. B.p. 237° (257°, 265–70°). D^{13} 0·7882, D_4^{20} 0·7859. Heat of comb. C_v 2452·1 Cal.

B,HCl: cryst. Sol. EtOH, Et_2O. Spar. sol. H_2O.

Isoamyliodide: tetra-isoamylammonium iodide. Prisms. M.p. 83°. D_4^{95} 1·0914. Spar. sol. H_2O.

Wallach, *Ann.*, 1905, **343**, 68.
Plimpton, *J. Chem. Soc.*, 1881, **39**, 332.
Hofmann, *Ann.*, 1851, **79**, 22.
Matter, D.R.P., 301,450, (*Chem. Zentr.*, 1918, I, 53).

Tri-isobutylamine

$[(CH_3)_2CH{\cdot}CH_2]_3N$

$C_{12}H_{27}N$ MW, 185

F.p. −24°. B.p. 184–6°. $D_4^{17\cdot3}$ 0·7711. $n_D^{17\cdot3}$ 1·42519. Non-misc. with H_2O. Heat of comb. C_v 1969 Cal.

Methochloroplatinate: m.p. 174°.
Ethochloroplatinate: m.p. 170°.
Propylchloroplatinate: m.p. 168°.
Butylchloroplatinate: m.p. 162°.

Ladenburg, *Ber.*, 1879, **12**, 949.
Malbot, *Ann. chim. phys.*, 1888, **13**, 493.

Tri-isobutyl phosphate.

See under Phosphoric Acid.

Tri-isobutyrin (*Glycerol tri-isobutyrate*)

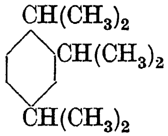
$CH_2{\cdot}O{\cdot}CO{\cdot}CH(CH_3)_2$
$CH{\cdot}O{\cdot}CO{\cdot}CH(CH_3)_2$
$CH_2{\cdot}O{\cdot}CO{\cdot}CH(CH_3)_2$

$C_{15}H_{26}O_6$ MW, 302

Liq. B.p. 282–4°, 173–6°/24 mm.

Guth, *Zeitschrift für Biologie*, 1903, **44**, 97.

1 : 2 : 4-Tri-isopropylbenzene



$C_{15}H_{24}$ MW, 204

Liq. B.p. 244°, 237°/736 mm., 113–14°/14 mm. D_{25}^{25} 0·8599. n_D^{25} 1·4896.

Slanina, Sowa, Nieuwland, *J. Am. Chem. Soc.*, 1935, **57**, 1549.
Kirrmann, Graves, *Bull. soc. chim.*, 1934, **1**, 1494.

Tri-isopropyl phosphate.
See under Phosphoric Acid.

Tri-isovalerin (*Isovalerin, glycerol tri-isovalerate*)

$CH_2·O·CO·CH_2·CH(CH_3)_2$
$CH·O·CO·CH_2·CH(CH_3)_2$
$CH_2·O·CO·CH_2·CH(CH_3)_2$

$C_{18}H_{32}O_6$ MW, 344

B.p. 330–5°/763 mm., 209·5–210·5°/27 mm. D^{20} 0·9984. n_D^{20} 1·43535.

Newman, Trikojus, Harker, *Chem. Abstracts*, 1926, **20**, 2658.

3 : 7 : 12-Triketocholanic Acid.
See Dehydrocholic Acid.

2 : 4 : 6 - Triketo - 1 : 1 - dimethylcyclohexane.
See Filicinic Acid.

2 : 4 : 6-Triketo-*n*-heptane.
See Diacetylacetone.

Triketohexane.
See Hexantrione.

Triketohydrindene Hydrate.
See Ninhydrin.

Triketopentane.
See Pentantrione.

1 : 3 : 5-Triketopimelic Acid (*Acetonedioxalic acid, xanthochelidonic acid*)

$HOOC·CO·CH_2·CO·CH_2·CO·COOH$

$C_7H_6O_7$ MW, 202

Amorph. Very unstable. Sol. H_2O, EtOH. Less sol. Et_2O. On standing ⟶ chelidonic acid. Forms stable salts.

Mono K salt: cryst. ppt. Mod. sol. cold H_2O.

Di-Et ester: $C_{11}H_{14}O_7$. MW, 258. Exists in mono- and di-enol forms. (i) *Mono-enol form*: prisms from EtOH. M.p. 104°. Sol. 60 parts Et_2O. Mod. sol. hot EtOH, C_6H_6. Warm ⟶ dienol form. (ii) *Dienol form*: yellow cryst. powder. M.p. 98°. Sol. 40 parts Et_2O. In solution is partially converted into monoenol form.

Lerch, *Monatsh.*, 1884, **5**, 375.
Claisen, *Ber.*, 1891, **24**, 116; D.R.P., 57,648.
Willstätter, Pummerer, *Ber.*, 1904, **37**, 3734.

2 : 3 : 4-Triketotetrahydroquinoline.
See Quinisatin.

Trilaurin (*Glycerol trilaurate, laurin*)

$CH_2·O·CO·[CH_2]_{10}·CH_3$
$CH·O·CO·[CH_2]_{10}·CH_3$
$CH_2·O·CO·[CH_2]_{10}·CH_3$

$C_{39}H_{74}O_6$ MW, 638

Found in laurel leaves, mahuba seeds and in fats of many other seeds. Needles from EtOH. M.p. 49° (45·6°). D_4^{60} 0·8944. n_D^{60} 1·44039. Sol. Et_2O, $CHCl_3$, C_6H_6, pet. ether. Spar. sol. cold EtOH. Heat of comb. C_v 4707 Cal.

Bömer, Engel, *Chem. Abstracts*, 1929, **23**, 4676.
André, *Compt. rend.*, 1927, **184**, 227.
Averil, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.

Trillin (3-α-*Glucoside of diosgenin*)

$C_{33}H_{52}O_8$ MW, 576

Occurs in *Trillium erectum* as the diglucoside, trillarin, m.p. 197–200°. Cryst. + $\frac{1}{2}H_2O$ from MeOH. M.p. 275–80°. EtOH-HCl ⟶ diosgenin.

Tetra-acetyl: cryst. from MeOH. M.p. 197°.

Marker, Krueger, *J. Am. Chem. Soc.*, 1940, **62**, 2548, 3349.

Trillogenin

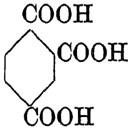
$C_{27}H_{48}O_4$ MW, 436

Steroid sapogenin occurring in *Trillium erectum*. Its unknown structure lacks the two characteristic inert oxygen atoms. Cryst. from MeOH. M.p. 206–10°. $[\alpha]_D^{24}$ −41·6°, $[\alpha]_{Hg}^{24}$ −54·3° in dioxan.

Tetra-acetyl: cryst. from MeOH.Aq. M.p. 102–3°. $[\alpha]_D^{24}$ 0°, $[\alpha]_{Hg}^{24}$ −3·5° in dioxan.

Lieberman, Chang, Barusch, Noller, *J. Am. Chem. Soc.*, 1942, **64**, 2581.

Trimellitic Acid (*Benzene* 1 : 2 : 4-*tricarboxylic Acid*)



$C_9H_6O_6$ MW, 210

Cryst. from AcOH or EtOH.Aq. M.p. 238° (229–30°). Sol. EtOH. Spar. sol. Me_2CO. Insol. $CHCl_3$, CCl_4, C_6H_6, CS_2. Sol. 20 parts boiling AcOH. k (first) = 3·2 × 10^{-3} at 25° : (second) = 1·1 × 10^{-4} at 25°.

1-*Me ester*: $C_{10}H_8O_6$. MW, 224. Exists in two forms; m.p. 177°, and m.p. 203·5–205·5°. Sol. H_2O, Et_2O, MeOH, hot AcOH (part. decomp.). Spar. sol. $CHCl_3$, CCl_4, CS_2. Very spar. sol. C_6H_6, pet. ether. k (first) = 1·8 × 10^{-3} at 25° : (second) = 7·8 × 10^{-5} at 25°.

2-*Me ester*: powder. M.p. 208°. Sol. hot H_2O. Mod. sol. Et_2O. Insol. $CHCl_3$, CCl_4, CS_2. k (first) = 2·6 × 10^{-3} at 25° : (second) = 9·9 × 10^{-5} at 25°.

4-*Me ester*: leaflets from H_2O. M.p. 145–7°. Sol. Et_2O. Mod. sol. H_2O. Insol. pet. ether. k (first) = 2·89 × 10^{-3} at 25° : (second) = 1·1 × 10^{-5} at 25°.

1 : 2-*Di-Me ester*: $C_{11}H_{10}O_6$. MW, 238. Needles from CS_2, C_6H_6 or Et_2O–pet. ether. M.p. 115·5–117°, solidifies and then remelts at 121°. B.p. above 200°/12 mm. $k = 3·4 \times 10^{-4}$ at 25°.

Tri-Me ester: $C_{12}H_{12}O_6$. MW, 252. Thick oil. F.p. — 13°. B.p. 194°/12 mm.

Tri-Et ester: $C_{15}H_{18}O_6$. MW, 294. B.p. 238°/28 mm.

1-*Amide*: $C_9H_7O_5N$. MW, 209. Yellowish cryst. from MeOH–C_6H_6. M.p. 185–6°. Sol. EtOH. Mod. sol. H_2O. Spar. sol. Et_2O, C_6H_6. k (first) $= 4·4 \times 10^{-4}$ at 25°.

2-*Amide*: cryst. from MeOH–C_6H_6. M.p. 199–200°. Sol. EtOH. Mod. sol. H_2O. Spar. sol. Et_2O, C_6H_6. k (first) $= 7·6 \times 10^{-4}$ at 25°.

4-*Amide*: cryst. from Et_2O–C_6H_6. M.p. 166°. Sol. H_2O, EtOH. Insol. $CHCl_3$, CCl_4, C_6H_6.

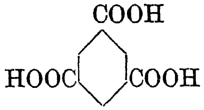
2-*Nitrile*: $C_9H_5O_4N$. MW, 191. Yellow amorph. mass. Sol. H_2O, EtOH, Et_2O.

Anhydride: anhydrotrimellitic acid. Needles. M.p. 162·5–163·5° (157°). B.p. 240–5°/14 mm. Sol. hot H_2O. *Me ester*: cryst. M.p. 94–9°.

Schultze, *Ann.*, 1908, **359**, 143.
Wegscheider, Perndanner, Auspitzer, *Monatsh.*, 1910, **31**, 1267.
Morgan, Coulson, *J. Chem. Soc.*, 1929, 2554.

Trimercaptobenzene.
See Trithiophloroglucinol.

Trimesic Acid (*Benzene*-1 : 3 : 5-*tricarboxylic acid*)



$C_9H_6O_6$ MW, 210

Needles or prisms from H_2O. M.p. about 380°. Very sol. EtOH. Sol. Et_2O. Sol. 40 parts H_2O at 23°. Heat of comb. C_v 768·5 Cal.

Mono-Me ester: $C_{10}H_8O_6$. MW, 224. Cryst. + H_2O from H_2O. M.p. anhyd. 205–8°. Sol. EtOH, Et_2O.

Tri-Me ester: $C_{12}H_{12}O_6$. MW, 252. Needles. M.p. 144°.

Tri-Et ester: $C_{15}H_{18}O_6$. MW, 294. Prisms or needles from EtOH. M.p. 133·5–134·5°. Very sol. Et_2O, MeOH, AcOEt, $CHCl_3$, CS_2. Sol. hot EtOH, AcOH, C_6H_6. Insol. H_2O.

Tri-isoamyl ester: $C_{24}H_{36}O_6$. MW, 420. Cryst. M.p. 28–9°. B.p. 278–80°/15 mm.

Trihydrazide: micro-plates from H_2O. Decomp. above 300°. Spar. sol. EtOH.

Wolff, Heip, *Ann.*, 1899, **305**, 153.
Wislicenus, v. Wrangell, *Ann.*, 1911, **381**, 372.
Ullmann, Uzbachian, *Ber.*, 1903, **36**, 1799.
v. Schaak, U.S.P., 1,706,639, (*Chem. Abstracts*, 1929, **23**, 2187).

3 : 4 : 5-Trimethoxy-1-allylbenzene.
See Elemicin.

2 : 4 : 6-Trimethoxybenzoylacetone.
See Eugenone.

3 : 4 : 5-Trimethoxycinnamic Acid.
See under Sinapic Acid.

6 : 7 : 8-Trimethoxycoumarin.
See under Fraxetin.

7 : 3′ : 5′-Trimethoxydelphinidin chloride.
See Hirsutidin chloride.

2 : 4 : 6-Trimethoxyisobutyrophenone.
See Conglomerone.

3 : 7 : 5′-Trimethoxy-3′ : 4′-methylenedioxyflavone.
See Kanugin.

3 : 4 : 5-Trimethoxyphenylacetic Acid.
See under Iridic Acid.

3 : 4 : 5-Trimethoxyphenyl-ethylamine.
See Mescaline.

2 : 4 : 5-Trimethoxy-1-propenylbenzene.
See Asarone.

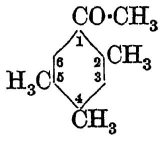
3 : 4 : 5-Trimethoxy-1-propenylbenzene.
See under Elemicin.

Trimethylacetaldehyde.
See Pivalic Aldehyde.

Trimethylacetic Acid.
See Pivalic Acid.

1 : 1 : 1-Trimethylacetone.
See Pinacolin.

2 : 4 : 5-Trimethylacetophenone (5-*Aceto-ψ-cumene*)



$C_{11}H_{14}O$ MW, 162

F.p. 11°. B.p. 246–7°, 137–8°/20 mm., 110°/7 mm. $D_4^{14·7}$ 1·0039. $n_D^{14·9}$ 1·541. Sol. EtOH, Et_2O, CS_2, AcOH, C_6H_6.

Oxime: cryst. from ligroin. M.p. 85–6°.

Semicarbazone: plates from EtOH. M.p. 204°.

Auwers, Köckritz, *Ann.*, 1907, **352**, 313.
Klages, Allendorff, *Ber.*, 1898, **31**, 1005.
Baker, Tweed, *J. Chem. Soc.*, 1941, 799.

2 : 4 : 6-Trimethylacetophenone (*Acetomesitylene*).

B.p. 240·5°/735 mm., 90°/3 mm. D^{20} 0·9754. n_D^{20} 1·5175.

Noller, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1893.
Meyer, Molz, *Ber.*, 1897, **30**, 1271.

ω-Trimethylacetophenone.
See tert.-Butyl phenyl Ketone.

Trimethylacrylic Acid (3-*Methyl*-2-*butylene*-2-*carboxylic acid*, 1-*isopropylidene-propionic acid*)

$$(CH_3)_2C{:}\overset{CH_3}{\overset{|}{C}}{\cdot}COOH$$

$C_6H_{10}O_2$ MW, 114

Long needles from H_2O. M.p. 70–1°. Sol. 20 parts H_2O at 19°. Sol. hot EtOH, Et_2O, $CHCl_3$, C_6H_6, ligroin. Spar. sol. hot H_2O. $k = 3{\cdot}9 \times 10^{-5}$ at 25°.

Et ester: $C_8H_{14}O_2$. MW, 142. B.p. 154–6° (153–7°/750 mm.). $D_4^{19\cdot3}$ 0·9072. $n_D^{19\cdot3}$ 1·430.

Nitrile: C_6H_9N. MW, 95. B.p. 155–7°. D^{18} 0·8447. Insol. H_2O.

Perkin, *J. Chem. Soc.*, 1896, **69**, 1479.
Henry, *Chem. Zentr.*, 1899, I, 195.
Merling, Welde, *Ann.*, 1909, **366**, 140.

1 : 1 : 3-Trimethyladipic Acid

$(CH_3)_2C{\cdot}COOH$
CH_2
$CH_3{\cdot}CH$
$CH_2{\cdot}COOH$

$C_9H_{16}O_4$ MW, 188

Cryst. from dry Et_2O. M.p. 80°.

Khuda, Ghosh, *J. Indian Chem. Soc.*, 1939, **16**, 293.

1 : 1 : 4-Trimethyladipic Acid (2-*Methylhexane*-2 : 5-*dicarboxylic acid*)

$(CH_3)_2C{\cdot}COOH$
CH_2
CH_2
$CH_3{\cdot}CH{\cdot}COOH$

$C_9H_{16}O_4$ MW, 188

Prisms from H_2O or dil. formic acid. M.p. 117° (114–15°). Mod. sol. dil. formic acid. Spar. sol. H_2O.

Auwers, Hessenland, *Ber.*, 1908, **41**, 1815.
Wallach, Kempe, *Ann.*, 1903, **329**, 91.

2 : 2 : 3-Trimethyladipic Acid

$CH_2{\cdot}COOH$
$(CH_3)_2C$
$CH_3{\cdot}CH$
$CH_2{\cdot}COOH$

$C_9H_{16}O_4$ MW, 188

Plates from di-isopropyl ether–EtOH. M.p. 127°.

Buchman, Sargent, *J. Org. Chem.*, 1942, **7**, 151.

Trimethylallantoin.
See Caffoline.

1 : 3 : 3-Trimethylallyl Alcohol (1 : 3-*Dimethylcrotonyl alcohol*, 1 : 3-*dimethyl*-2-*butenol*-1)

CH_3
$(CH_3)_2C{:}CH{\cdot}CH{\cdot}OH$

$C_6H_{12}O$ MW, 100

d-.

B.p. 129°/760 mm., 43°/17 mm. D_4^{20} 0·8436. n_D^{17} 1·4297. $[\alpha]_{5461}^{22}$ + 11·04°.

Acid phthalate: needles from pet. ether. M.p. 44°. $[\alpha]_{5461}$ + 16·7° in $CHCl_3$, + 22·2° in C_6H_6. *Brucine salt*: needles from Me_2CO. M.p. 144°. $[\alpha]_{5461}$ − 23·0° in $CHCl_3$.

Acetyl: b.p. 51°/15 mm. n_D^{20} 1·4202. $[\alpha]_{5461}^{22}$ − 4·72°.

Benzoyl: b.p. 139°/17 mm. n_D^{20} 1·5047. $[\alpha]_{5461}^{20}$ + 35·71°.

l-.

B.p. 129°/760 mm., 43°/18 mm. n_D^{17} 1·4298. $[\alpha]_{5461}^{20}$ − 2·60°.

Acid phthalate: needles from pet. ether. M.p. 44°. $[\alpha]_{5461}$ − 14·2° in CS_2. *Strychnine salt*: needles from $CHCl_3$ + 4 vols. Me_2CO. $[\alpha]_{5461}$ − 24·89°.

dl-.

B.p. 131–3° (125–35°).

Acid phthalate: prisms from Et_2O–pet. ether. M.p. 81·5°.

Acetyl: b.p. 50°/15 mm. n_D^{20} 1·4201.

Benzoyl: b.p. 138°/19 mm. n_D^{20} 1·5045.

Duveen, Kenyon, *J. Chem. Soc.*, 1936, 1451.

Trimethylamine

$(CH_3)_3N$

C_3H_9N MW, 59

Liq. with fishy odour. M.p. − 117·2°. B.p. 3·2–3·8°/746·6 mm. D_4^0 0·6709, D_4^{-79} 0·7537. Heat of comb. C_v 592·1 Cal. $k = 7{\cdot}4 \times 10^{-5}$ at 25°. Crit. temp. 160·5°. Crit. press. 41 atm. Misc. with H_2O, EtOH. Heat with ethylene chlorohydrin ⟶ choline chloride.

B,11H₂O: m.p. 5·34°.

B,HCl: needles from EtOH. M.p. 277–8° decomp. Sublimes at 200°. Sol. EtOH. Mod. sol. $CHCl_3$. Insol. Et_2O.

B,HBr: prisms from EtOH. M.p. 243–5°.

B,HI: prisms from 95% EtOH. M.p. 263°. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O, $CHCl_3$.

B,HAuCl₄: yellow prisms from EtOH. M.p. 287° (237°).

Picrate: pale yellow prisms. M.p. 216°.

Oxide: *see* Trimethylamine oxide.

Adams, Brown, *Organic Syntheses*, Collective Vol. I, 514.
Schmitz, D.R.P., 270,260, (*Chem. Zentr.*, 1914, I, 830).
Koeppen, *Ber.*, 1905, **38**, 883.
Schmidt, *Ann.*, 1892, **267**, 267.
Dreyfus, B.P. 398,502, (*Chem. Abstracts*, 1934, **28**, 1357).

Trimethylamine oxide (*Trimethyloxamine*)

$(CH_3)_3NO$

C_3H_9ON MW, 75

Widely distributed in fish and animal tissues. Needles + $2H_2O$ from H_2O. M.p. 255–7° (208°), anhyd. 96°. Sol. H_2O, MeOH. Less sol. EtOH. Heat at 180° ⟶ formaldehyde + trimethylamine. Reacts strongly alkaline. Does not reduce Fehling's.

B,HCl: needles from EtOH. M.p. 218°. Sol. H_2O, hot MeOH.

B,HI: prisms from EtOH. M.p. 130° decomp. Sol. H_2O, EtOH. Insol. Et_2O.

B,HAuCl$_4$: yellow cryst. M.p. 200°.

B_2,H_2PtCl_6: m.p. 228–9° decomp.

Picrate: yellow needles. M.p. 196–8°.

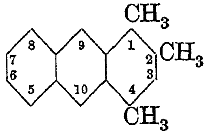
Hoppe-Seyler, Schmidt, *Zeitschrift für Biologie*, 1928, **87**, 59.
Meisenheimer, *Ann.*, 1913, **397**, 287.
Dunstan, Goulding, *J. Chem. Soc.*, 1899, **75**, 792, 1005.

Trimethylamine-tricarboxylic Acid.
See Triglycolamidic Acid.

Trimethyl-ω-aminoethylcyclopentene.
See Camphylamine.

2 : 4 : 6-Trimethylanisole.
See under Mesitol.

1 : 2 : 4-Trimethylanthracene



$C_{17}H_{16}$ MW, 220

Cryst. M.p. 243°.

Gresly, *Ann.*, 1886, **234**, 239.
Wende, *Ber.*, 1887, **20**, 868.
I.G., B.P. 253,911, (*Chem. Abstracts*, 1927, **21**, 2478).

1 : 2 : 10-Trimethylanthracene.
Yellow needles from hexane. M.p. 88–90°.
Picrate: brown needles from EtOH. M.p. 138·5–139·5°.
sym.-*Trinitrobenzene add. comp.*: scarlet needles from EtOH. M.p. 169·6–170·2°.

Fieser, Webber, *J. Am. Chem. Soc.*, 1940, **62**, 1361.

1 : 3 : 6-Trimethylanthracene.
Cryst. M.p. 222°. Sol. Me_2CO, CS_2, C_6H_6. Mod. sol. Et_2O, AcOH. Spar. sol. EtOH, ligroin.

Elbs, *J. prakt. Chem.*, 1890, **41**, 142.

1 : 3 : 10-Trimethylanthracene.
Yellow cryst. from pet. ether. M.p. 100°.

Barnett, Hewett, *Ber.*, 1931, **64**, 1577.

1 : 4 : 9-Trimethylanthracene.
Pale yellow cryst. from MeOH. M.p. 81°. Sols. show strong fluor.

Barnett, Low, *Ber.*, 1931, **64**, 53.

1 : 9 : 10-Trimethylanthracene.
Yellow needles from EtOH. M.p. 90–2°.
Picrate: black needles from EtOH. M.p. 142–4°.

Sandin, Kitchen, *J. Am. Chem. Soc.*, 1945, **67**, 1306.

2 : 3 : 6-Trimethylanthracene.
Found in low-temperature coal tar. Pale yellow flakes with bluish fluor. from boiling AcOH. M.p. 255°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2555; *J. Soc. Chem. Ind.*, 1934, **53**, 71T.

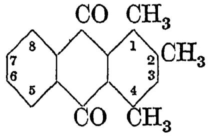
2 : 3 : 9-Trimethylanthracene.
Pale yellow cryst. from AcOEt. M.p. 125°. Sols. show strong fluor.

Barnett, Morrison, *Ber.*, 1931, **64**, 538.

2 : 9 : 10-Trimethylanthracene.
Yellow needles. M.p. 93–6°.
Picrate: black needles from C_6H_6. M.p. 162–162·5°.

Sandin, Kitchen, *J. Am. Chem. Soc.*, 1945, **67**, 1306.
Bachman, Chemerda, *J. Org. Chem.*, 1939, **4**, 587.

1 : 2 : 4-Trimethylanthraquinone



$C_{17}H_{14}O_2$ MW, 250

Yellow needles from xylene–EtOH. M.p. 162–3°. Sol. Et_2O, C_6H_6. Spar. sol. EtOH.

Gresly, *Ann.*, 1886, **234**, 241.
Elbs, *J. prakt. Chem.*, 1890, **41**, 123.

1 : 3 : 6-Trimethylanthraquinone.
Pale yellow needles from AcOH. M.p. 192°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.
Elbs, *J. prakt. Chem.*, 1890, **41**, 143.

1 : 3 : 7-Trimethylanthraquinone.
Pale yellow needles from AcOH. M.p. 128–9°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.

1 : 4 : 5-Trimethylanthraquinone.
Yellow needles from AcOH. M.p. 146–7°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.

1 : 4 : 6-Trimethylanthraquinone.
Yellow needles from AcOH. M.p. 143°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.
Cf. Elbs, *J. prakt. Chem.*, 1890, **41**, 142.

2 : 3 : 6-Trimethylanthraquinone.
Very pale yellow needles from boiling AcOH. M.p. 240°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2555.
Fieser, Seligman, *J. Am. Chem. Soc.*, 1934, **56**, 2695.

Trimethylarsine (*Arsenic trimethyl*)

$As(CH_3)_3$

C_3H_9As MW, 120

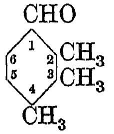
Colourless liq. B.p. about 70°, (48–51°). Absorbs O from the air to give the oxide. Combines direct with S and halogens. Very poisonous.

B,HgCl₂: needles from hot H_2O. M.p. 224–6°.

B,2HgCl₂: plates from hot aq. $HgCl_2$. M.p. 264–5° decomp.

Hibbert, *Ber.*, 1906, **39**, 161.
Gryszkiewicz-Trochimowski, Zambrzycki, *Chem. Abstracts*, 1927, **21**, 3612.
Challenger, Higginbottom, Ellis, *J. Chem. Soc.*, 1933, 95.
Challenger, Higginbottom, *Biochem. J.*, 1935, **29**, 1757.

2 : 3 : 4-Trimethylbenzaldehyde (*4-Aldehydohemimellitene*)



$C_{10}H_{12}O$ MW, 148

M.p. 7–8°. B.p. 121·5°/11 mm.

Oxime: cryst. from EtOH.Aq. M.p. 131–2°.

Smith, Agre, *J. Am. Chem. Soc.*, 1938, **60**, 651.

2 : 3 : 6-Trimethylbenzaldehyde (*3-Aldehydo-ψ-cumene*).

Colourless liq. B.p. 113–14°/10 mm.

Semicarbazone: cryst. from EtOH. M.p. 167–9°.

Oxime: cryst. from EtOH.Aq. M.p. 124–6°.

Smith, Nichols, *J. Org. Chem.*, 1941, **6**, 501.

2 : 4 : 5-Trimethylbenzaldehyde (*5-Aldehydo-ψ-cumene, duryl aldehyde*).

Plates from EtOH. M.p. 43·5° (42°). B.p. 243°, 121°/10 mm. Turns yellow in air.

Oxime: needles from ligroin. M.p. 102°.

Semicarbazone: prisms from EtOH. M.p. 243–4°.

Hydrazone: cryst. M.p. 70°. B.p. 165–6°.

Picrate: yellow cryst. powder. M.p. 170–1°.

Phenylhydrazone: leaflets from AcOH. M.p. 127°.

Gattermann, *Ann.*, 1906, **347**, 375.
Auwers, Köckritz, *Ann.*, 1907, **352**, 310.
Smith, Nichols, *J. Org. Chem.*, 1941, **6**, 501.
Niedzielski, Nord, *J. Org. Chem.*, 1943, **8**, 148.

2 : 4 : 6-Trimethylbenzaldehyde (*β-Isoduryl aldehyde, mesityl aldehyde, aldehydomesitylene*).

B.p. 237–40°, 192°/50 mm. Fuming HNO_3 + AcOH ⟶ 3-nitro-deriv., m.p. 61°.

Di-Me acetal: b.p. 242–3°/741 mm.

Oxime: *syn*-., needles. M.p. 180–1°. Less sol. than *anti*-form in ord. org. solvents. *Anti*-. Prisms from Et_2O. M.p. 124°. Heat with dil. HCl ⟶ *syn*.-form ⟶ 2 : 4 : 6-trimethylbenzonitrile. *Acetate*: m.p. 68°.

Semicarbazone: cryst. from EtOH. M.p. 185–8°.

Phenylhydrazone: cryst. from EtOH.Aq. M.p. 74°.

Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2797; 1931, 1170.
Wenzel, *Monatsh.*, 1914, **35**, 968.
Fischer, Giebe, *Ber.*, 1898, **31**, 548.
Hantzsch, Lucas, *Ber.*, 1895, **28**, 747.
Barnes, *Organic Syntheses*, 1941, XXI, 110.
Fuson *et al.*, *Organic Syntheses*, 1943, XXIII, 57.

3 : 4 : 5-Trimethylbenzaldehyde (*5-Aldehydohemimellitene*).

Needles from dil. EtOH. M.p. 52°.

Krömer, *Ber.*, 1891, **24**, 2413.

1 : 2 : 3-Trimethylbenzene.
See Hemimellitene.

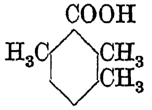
1 : 2 : 4-Trimethylbenzene.
See ψ-Cumene.

1 : 3 : 5-Trimethylbenzene.
See Mesitylene.

2 : 3 : 4-Trimethylbenzoic Acid.
See Prehnitylic Acid.

2 : 3 : 5-Trimethylbenzoic Acid.
See γ-Isodurylic Acid.

2 : 3 : 6-Trimethylbenzoic Acid



$C_{10}H_{12}O_2$ MW, 164

Needles from H_2O or pet. ether. M.p. 110–11°.

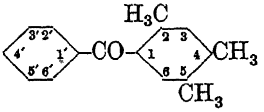
Lapworth, Wechsler, *J. Chem. Soc.*, 1907, **91**, 994.
Smith, Agre, *J. Am. Chem. Soc.*, 1938, **60**, 653.

2 : 4 : 5-Trimethylbenzoic Acid.
See Durylic Acid.

2 : 4 : 6-Trimethylbenzoic Acid.
See β-Isodurylic Acid.

3 : 4 : 5-Trimethylbenzoic Acid.
See α-Isodurylic Acid.

2 : 4 : 5 - Trimethylbenzophenone (*5-Benzoyl-ψ-cumene*)



$C_{16}H_{16}O$ MW, 224

B.p. 328°/760 mm., 211°/23 mm. D_4^{18} 1·0332.

Klages, Allendorff, *Ber.*, 1898, **31**, 1001.
Elbs, *J. prakt. Chem.*, 1887, **35**, 491.

2 : 4 : 6-Trimethylbenzophenone (*Benzoylmesitylene*).

Prisms from $EtOH-Et_2O$. M.p. 35·5°. B.p. 318–20° (326·5–327°/777 mm.), 189°/17 mm. Sol. Me_2CO, $CHCl_3$, AcOH, pet. ether.

Louise, *Ann. chim. phys.*, 1885, **6**, 202.
Elbs, *J. prakt. Chem.*, 1887, **35**, 486.
Kharasch, Morrison, Urry, *J. Am. Chem. Soc.*, 1944, **66**, 368.

2 : 4 : 4′-Trimethylbenzophenone (4-p-*Toluyl*-m-*xylene*).

Pale yellow viscous oil. B.p. 340°, 169°/4 mm. Does not react with phenylhydrazine or semicarbazide. Reacts only very slowly with hydroxylamine. Hot conc. $H_2SO_4 \longrightarrow$ *p*-toluic acid + *m*-xylene.

Oxime: cryst. from EtOH. M.p. 132°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2209.

2 : 5 : 4′-Trimethylbenzophenone (2-p-*Toluyl*-p-*xylene*).

Rhombic plates from EtOH. M.p. 54°. B.p. 337°/760 mm., 202°/23 mm. Unreactive towards usual ketonic reagents.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2209.

2 : 6 : 2′-Trimethylbenzophenone (2-o-*Toluyl*-m-*xylene*).

Cryst. M.p. 69°. Does not react with 2 : 4-dinitrophenylhydrazine.

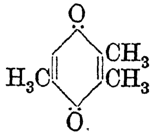
Coops *et al.*, *Rec. trav. chim.*, 1940, **59**, 1111.

2 : 3′ : 4′-Trimethylbenzophenone (4-o-*Toluyl*-o-*xylene*).

Cryst. from pet. ether. M.p. 55–8°. B.p. 192–7°/15 mm.

Baker *et al.*, *J. Chem. Soc.*, 1945, 30.

2:3:5-Trimethyl-*p*-benzoquinone (2:3:5-*Trimethylquinone*, *ψ-cumoquinone*)



$C_9H_{10}O_2$ MW, 150

Yellow needles from Et_2O. M.p. 32° (11°). Volatile in steam.

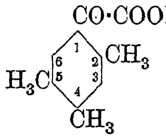
1-*Oxime*: golden-yellow needles from EtOH. M.p. 184°. Possesses strong quinone odour.

4-*Oxime*: yellow needles. M.p. 172°. Odourless.

1-*Semicarbazone*: yellow cryst. M.p. 252–3°.

Noelting, Baumann, *Ber.*, 1885, **18**, 1152.
Nietzki, Schneider, *Ber.*, 1894, **27**, 1430.

2 : 4 : 5-Trimethylbenzoylformic Acid (2 : 4 : 5-*Trimethylphenylglyoxylic acid*)



$C_{11}H_{12}O_3$ MW, 192

Needles. M.p. 76° (61–2°). Sol. EtOH, Et_2O, $CHCl_3$, hot H_2O.

Et ester: $C_{13}H_{16}O_3$. MW, 220. B.p. 175–6°/10 mm.

Claus, *J. prakt. Chem.*, 1890, **41**, 509.
Bouveault, *Bull. soc. chim.*, 1897, **17**, 363, 369.

2 : 4 : 6-Trimethylbenzoylformic Acid (2 : 4 : 6-*Trimethylphenylglyoxylic acid, mesitylglyoxylic acid*).

Pale yellow cryst. from CS_2. M.p. 117–18° (114–17°). Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. CS_2, ligroin. $k = 5{\cdot}27 \times 10^{-2}$ at 25°. Decomp. on dist.

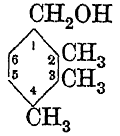
Me ester: $C_{12}H_{14}O_3$. MW, 206. B.p. 253–5° (273–5°), 170°/100 mm.

Et ester: b.p. 265–7°, 164–5°/10 mm. Turns brown on standing.

Hydrazone: needles + H_2O. M.p. 200° decomp. Sol. EtOH. Mod. sol. boiling H_2O. Insol. Et_2O, $CHCl_3$, C_6H_6.

Bouveault, *Bull. soc. chim.*, 1897, **17**, 371.
Claus, *J. prakt. Chem.*, 1890, **41**, 504.
Wenzel, *Monatsh.*, 1914, **35**, 948.

2 : 3 : 4-Trimethylbenzyl Alcohol



$C_{10}H_{14}O$ MW, 150

Needles from pentane. M.p. 49–50°. B.p. 110–30°/0·5 mm. Sol. usual org. solvents.

Reichstein, Cohen, Ruth, Meldahl, *Helv. Chim. Acta*, 1936, **19**, 416.

2 : 3 : 6-Trimethylbenzyl Alcohol.

Cryst. from pet. ether. M.p. 83·5–85°.

Smith, Agre, *J. Am. Chem. Soc.*, 1938, **60**, 653.

2 : 4 : 5-Trimethylbenzyl Alcohol.

Needles from EtOH. M.p. 168°.

Krömer, *Ber.*, 1891, **24**, 2411.

2 : 4 : 6-Trimethylbenzyl Alcohol (*Mesitylcarbinol*).

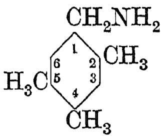
Needles. M.p. 88–9°. B.p. 140–1°/15 mm. Sol. usual org. solvents.

Me ether: b.p. 109–10°/15 mm.

Et ether: b.p. 114–15°/14 mm.

Carré, *Bull. soc. chim.*, 1910, **7**, 842.
Nauta, Dienske, *Rec. trav. chim.*, 1936, **55**, 1000.

2 : 4 : 5-Trimethylbenzylamine (ω-*Amino-1 : 2 : 4 : 5-tetramethylbenzene*)



$C_{10}H_{15}N$ MW, 149

Two compounds of this structure have been described in the literature.

(i) Needles from EtOH.Aq. M.p. 64·5°. Sol. EtOH, $CHCl_3$. Insol. cold H_2O.

B,HCl: prisms or needles. M.p. 240–2° decomp.

Krömer, *Ber.*, 1891, **24**, 2409.

(ii) Leaflets from MeOH.Aq. M.p. 52°. Sol. usual solvents. Spar. sol. cold H_2O.

B,HCl: plates. M.p. 275–6°. Sol. hot H_2O.

Acetyl: needles. M.p. 143·5°. Mod. sol. EtOH, C_6H_6. Spar. sol. H_2O.

Willstätter, Kubli, *Ber.*, 1909, **42**, 4156.

2 : 4 : 6-Trimethylbenzylamine (ω-*Amino-1 : 2 : 4 : 6-tetramethylbenzene*).

B,HCl: needles from H_2O. M.p. 315°.

N-*Benzyl*: cryst. from EtOH. M.p. 153·5–154°.

N-*Acetyl*: cryst. from EtOH. M.p. 186·5–187°.

Fuson, Denton, *J. Am. Chem. Soc.*, 1941, **63**, 656.

3 : 4 : 5-Trimethylbenzylamine (ω-*Amino-1 : 2 : 3 : 5-tetramethylbenzene*).

Leaflets from boiling H_2O. M.p. 123°.

B,HCl: needles from EtOH. M.p. 270°.

B,HAuCl$_4$: red prisms. M.p. 162–5° decomp.

B_2,H_2PtCl_6: rhombohedra. M.p. 219–20° decomp.

Picrate: yellow needles from EtOH.Aq. M.p. 239–40° decomp.

Krömer, *Ber.*, 1891, **24**, 2411.

1 : 8 : 8-Trimethylbicyclo-[1.2.3]-octanone-2.

See Homocamphor.

1 : 8 : 8-Trimethylbicyclo-[1.2.3]-octanone-3.

See Homoepicamphor.

Trimethyl borate (*Methyl borate*)

$B(OCH_3)_3$

$C_3H_9O_3B$ MW, 104

B.p. 55–6° (65°). D^0 0·940, D^{20} 0·915. Burns with green flame. Hyd. by H_2O.

Schiff, *Ann., Suppl.*, 1867, **5**, 183.
Gasselin, *Ann. chim. phys.*, 1894, **3**, 22.
Cohn, *Pharm. Zentralhalle*, 1911, **52**, 480.

1 : 1 : 4-Trimethylbutadiene-1 : 3.

See 2-Methyl-2 : 4-hexadiene.

1 : 2 : 4-Trimethylbutadiene-1 : 3.

See 3-Methyl-2 : 4-hexadiene.

1 : 1 : 1-Trimethylbutane.

See 2 : 2-Dimethylpentane.

1 : 5 : 5-Trimethyl-6-α-butenylcyclohexene.

See Ionane.

Trimethylbutylene.

See Dimethylpentene.

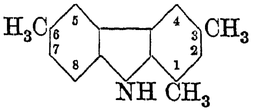
Trimethyl-*n*-butylmethane.

See 2 : 2-Dimethyl-*n*-hexane.

Trimethylcaffolide.

See under Caffolide.

1 : 3 : 6-Trimethylcarbazole



$C_{15}H_{15}N$ MW, 209

Cryst. from pet. ether. M.p. 126°. Sol. EtOH, AcOH, C_6H_6.

N-*Nitroso*: yellow needles from pet. ether. M.p. 139°.

Fries, Böker, Wallbaum, *Ann.*, 1934, **509**, 94.

1 : 3 : 7-Trimethylcarbazole.

Leaflets from EtOH. M.p. 119°.

Picrate: deep red cryst. M.p. 177°.

Borsche, *Ann.*, 1908, **359**, 77.

1 : 4 : 8-Trimethylcarbazole.

M.p. 153–4°.

Longo, Pirona, *Gazz. chim. ital.*, 1947, **77**, 127.

2 : 4 : 6-Trimethylcarbazole.

Prisms from pet. ether. M.p. 198°.

Fries, Böker, Wallbaum, *Ann.*, 1934, **509**, 98.

Trimethylcarbinol.

See tert.-Butyl Alcohol.

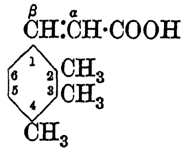
1 : 2 : 2-Trimethyl-3-carboxycyclopentylacetic Acid.

See Homoepicamphoric Acid.

1 : 2 : 2-Trimethyl-3-carboxymethylcyclopentane-1-carboxylic Acid.

See Homocamphoric Acid.

2 : 3 : 4-Trimethylcinnamic Acid



$C_{12}H_{14}O_2$ MW, 190

Cryst. from AcOH.Aq. M.p. 207–8°.

Smith, Agre, *J. Am. Chem. Soc.*, 1938, **60**, 681.

2 : 4 : 5-Trimethylcinnamic Acid.

Plates from EtOH.Aq. M.p. 154–5°.

Smith, Tawney, *J. Am. Chem. Soc.*, 1934, **56**, 2169.
Smith, Denyes, *J. Am. Chem. Soc.*, 1936, **58**, 306.

2 : 4 : 6-Trimethylcinnamic Acid.

Needles from EtOH.Aq. or AcOH.Aq. M.p. 176°. Sol. EtOH, Et_2O, AcOH, C_6H_6, toluene. Spar. sol. $CHCl_3$, CCl_4, CS_2.

Et ester: $C_{14}H_{18}O_2$. MW, 218. Cryst. from EtOH.Aq. or pet. ether. M.p. 40°. B.p. 170°/16 mm. Very sol. EtOH, Et_2O, AcOH, AcOEt, $CHCl_3$, C_6H_6, ligroin, pet. ether.

Böck, Lock, Schmidt, *Monatsh.*, 1934, **64**, 413.

2 : 4 : β-Trimethylcinnamic Acid.

Cryst. from pet. ether. M.p. 82–5°. B.p. 175–9°/12 mm., 156°/0·4 mm.

Et ester: $C_{14}H_{18}O_2$. MW, 218. B.p. 160–70°/20 mm.

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2540.

Mazurewitsch, *Chem. Zentr.*, 1914, I, 1999.

Wagner-Jauregg *et al.*, *Ber.*, 1943, **76**, 116.

2 : 5 : β-Trimethylcinnamic Acid.

Cis:

Oil.

Et ester: b.p. 150–4°/20 mm., 148–50°/12 mm.

Trans:

Needles from pet. ether. M.p. 68°. Sol. most solvents.

Auwers, Risse, *Ann.*, 1933, **502**, 294.

Ruzicka, Ehmann, Hefti, Altana, *Helv. Chim. Acta*, 1932, **15**, 157.

2 : α : β-Trimethylcinnamic Acid.

Et ester: pale yellow oil. B.p. 128–32°/11 mm.

Ruzicka, Ehmann, Hartnagel, Hausschild, *Helv. Chim. Acta*, 1932, **15**, 150.

4 : α : β-Trimethylcinnamic Acid.

Leaflets from EtOH.Aq. M.p. 163°.

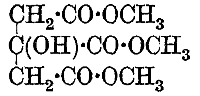
Et ester: b.p. 149–149·5°/10 mm. (141–141·5°/11 mm.). $D_4^{18\cdot7}$ 1·0024. $n_D^{18\cdot4}$ 1·519.

Auwers, *Ann.*, 1917, **413**, 278.

Rupe, Steiger, Fiedler, *Ber.*, 1914, **47**, 73.

Campbell, Soffer, *J. Am. Chem. Soc.*, 1942, **64**, 424.

Trimethyl citrate



$C_9H_{14}O_7$ MW, 234

M.p. 78–9°. B.p. 283–7° decomp. ⟶ aconitic acid trimethyl ester.

2-*Acetyl*: 2-acetoxytricarballylic trimethyl ester. B.p. 280–2° decomp., 171°/15 mm.

Me ether: $C_{10}H_{16}O_7$. MW, 248. B.p. 159–60°/12 mm. Sol. EtOH, Et_2O.

Hunäus, *Ber.*, 1876, **9**, 1750.

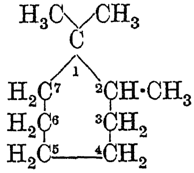
Anschütz, *Ann.*, 1903, **327**, 229.

Saint-Evre, *Ann.*, 1847, **60**, 325.

Trimethylcycloheptadienone.

See Eucarvone.

1 : 1 : 2-Trimethylcycloheptane



$C_{10}H_{20}$ MW, 140

Liq. B.p. 104–5°/100 mm. D_4^{20} 0·8243. n_D^{20} 1·4527.

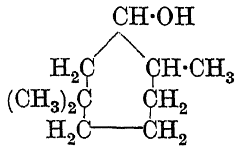
Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 430.

1 : 1 : 4-Trimethylcycloheptane (*Eucarvane*).

Liq. B.p. 162–3°/720 mm. D_4^{20} 0·8011. n_D^{20} 1·4420.

Ruzicka, Siedel, *Helv. Chim. Acta*, 1936, **19**, 430.

2 : 6 : 6-Trimethylcycloheptanol



$C_{10}H_{20}O$ MW, 156

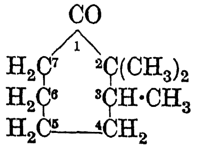
Liq. B.p. 216°. D^{24} 0·9096. n_D 1·4639.

Phenylurethane: m.p. 76°.

Wallach, *Ann.*, 1914, **403**, 90.

Wallach, Köhler, *Ann.*, 1905, **339**, 96, 106.

2 : 2 : 3-Trimethylcycloheptanone



$C_{10}H_{18}O$ MW, 154

Liq. with mint-like odour. B.p. 90°/14 mm. D_4^{20} 0·9211. n_D^{20} 1·4622.

Semicarbazone: cryst. from MeOH. M.p. 172–172·5°.

2 : 4-*Dinitrophenylhydrazone*: red needles from MeOH. M.p. 162–162·5°.

Naef, D.R.P., 580,713, (*Chem. Abstracts*, 1934, **28**, 1716).

Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 430.

Naves, *Helv. Chim. Acta*, 1948, **31**, 156.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1948, **31**, 427.

2 : 2 : 6-Trimethylcycloheptanone.

Liq. B.p. 191°. $D_4^{14\cdot5}$ 0·878. $n_D^{14\cdot5}$ 1·4421.

Semicarbazone: m.p. 142°.

Colonge, Dumont, *Bull. soc. chim.*, 1947, **14**, 44.

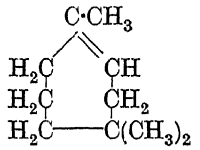
2 : 3 : 7-Trimethylcycloheptanone.
Liq. B.p. 204°. D_4^{23} 0·902. n_D^{23} 1·4540.
Semicarbazone : m.p. 178°.

Colonge, Dumont, *Bull. soc. chim.*, 1947, **14**, 42.

2 : 6 : 6-Trimethylcycloheptanone (*Tetrahydroeucarvone*).
Liq. with peppermint odour. B.p. 208–9°, 85·5°/12 mm. D^{18} 0·9095. n_D^{18} 1·4568.
Semicarbazone : exists in two forms. (i) Cryst. M.p. 191–2°. Spar. sol. AcOEt. (ii) Needles. M.p. 161–3°. Sol. AcOEt.

Wallach, *Ann.*, 1911, **381**, 67; 1914, **403**, 89.
Wallach, Köhler, *Ann.*, 1905, **339**, 107.

1 : 4 : 4-Trimethylcycloheptene (*Eucarvene*)

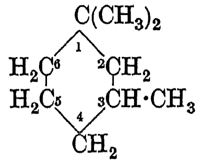


$C_{10}H_{18}$ MW, 138
Liq. B.p. 161–5°/720 mm. D_4^{20} 0·8185. n_D^{20} 1·4561.

Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 431.
Barbier, *Helv. Chim. Acta*, 1940, **23**, 528.

2 : 6 : 6-Trimethyl-1 : 3-cyclohexadiene-1-carboxylic Acid.
See Safranic Acid.

1 : 1 : 3-Trimethylcyclohexane



C_9H_{18} MW, 126
B.p. 137–8°. D_4^{25} 0·7868. n_D^{20} 1·4362. Heat of comb. C_v 1406 Cal.

Auwers, *Ann.*, 1920, **420**, 109.
Knoevenagel, *Ann.*, 1897, **297**, 202.

1 : 2 : 3-Trimethylcyclohexane (*Hexahydrohemimellitene*).
Cis :
B.p. 144–6°/755 mm. D_4^{20} 0·7930. n_{He}^{20} 1·43682.
Trans :
B.p. 142–143·5°/762 mm. D_4^{20} 0·7914. n_{He}^{20} 1·43582.

Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 552.

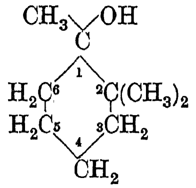
1 : 2 : 4-Trimethylcyclohexane (*Hexahydro-ψ-cumene*).
Found in petroleum from Maki, Echigo Province.
Cis :
B.p. 146° (142°). D_4^{20} 0·786. n_D^{20} 1·43209.
Trans :
B.p. 140–1°, 138·5–139·5°/755 mm. D_4^{20} 0·7813. n_{He}^{20} 1·43121.

Iimori, Kikuchi, *Chem. Abstracts*, 1928, **22**, 496.
Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 553.

1 : 3 : 5-Trimethylcyclohexane (*Hexahydromesitylene*).
Cis :
B.p. 140·0–140·5°/752 mm. D_4^{20} 0·7773. n_H^{20} 1·43010.
Trans :
B.p. 138·5–139·0°/754 mm. D_4^{20} 0·7720. n_H^{20} 1·42740.

Adams, Marshall, *J. Am. Chem. Soc.*, 1928, **50**, 1970.
Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 554.

1 : 2 : 2-Trimethylcyclohexanol



$C_9H_{18}O$ MW, 142
Oil. B.p. 81·4–81·8°/20 mm., 75·8°/16 mm. $D_4^{18·4}$ 0·9274. n_D^{20} 1·4682.
Hydrate, $\frac{1}{2}H_2O$: cryst. M.p. 41°.

Auwers, Lange, *Ann.*, 1913, **401**, 322.
Colonge, Duroux, *Bull. soc. chim.*, 1940, **7**, 459.

1 : 2 : 6-Trimethylcyclohexanol.
B.p. 78°/23 mm. D_4^{15} 0·9126. n_D^{15} 1·4598.

Zelinsky, Dvorjantchik, *Bull. soc. chim.*, 1904, **32**, 746.

1 : 3 : 3-Trimethylcyclohexanol.
Prisms. M.p. 74° (72·5°). Sol. most org. solvents.

Crossley, Gilling, *J. Chem. Soc.*, 1910, **97**, 2220.

1 : 3 : 5-Trimethylcyclohexanol.
B.p. 181°, 82–3°/19 mm. $D_4^{16·8}$ 0·8876. $n_D^{16·3}$ 1·454.

Wallach, *Ann.*, 1913, **396**, 284.

1 : 4 : 4-Trimethylcyclohexanol.
Needles. M.p. 58°. B.p. 79–80°/15 mm., 75°/11 mm.

Auwers, Lange, *Ann.*, 1913, **401**, 317.

2 : 2 : 3-Trimethylcyclohexanol.
B.p. 85–7°/15 mm.

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 159.

2 : 2 : 5-Trimethylcyclohexanol.
See Pulenol.

2 : 2 : 6-Trimethylcyclohexanol.
Cryst. from EtOH or pet. ether. M.p. 51°. B.p. 186–7°/753 mm., 87°/28 mm. D_4^{20} 0·9128. n_D^{20} 1·4600.

Haller, *Compt. rend.*, 1913, **157**, 181.
Masson, *Compt. rend.*, 1912, **154**, 518.

2 : 3 : 3-Trimethylcyclohexanol.
Needles. M.p. 28°. B.p. 197°. Very sol. org. solvents.

Crossley, Renouf, *J. Chem. Soc.*, 1911, **99**, 1108; 1915, **107**, 607.

2 : 3 : 5-Trimethylcyclohexanol.
Liq. B.p. 196–7°. D^{25} 0·9002. n_D^{25} 1·4572.
α-Naphthylurethane : m.p. 148–9°.

Ungnade, McLaren, *J. Am. Chem. Soc.*, 1944, **66**, 121.

2 : 3 : 6-Trimethylcyclohexanol.
B.p. 193–5°/747 mm. D_4^{17} 0·9119.

Zelinsky, Reformatski, *Ber.*, 1895, **28**, 2945.

2 : 4 : 4-Trimethylcyclohexanol.
Viscous oil. B.p. 192–3°.

Wallach, Scheunert, *Ann.*, 1902, **324**, 106.

2 : 4 : 5-Trimethylcyclohexanol.
Cis :
B.p. 191–3°, 84°/17 mm.
Phenylurethane : cryst. from EtOH. M.p. 83·5°.
Trans :
B.p. 196°, 112°/35 mm.
Phenylurethane : cryst. from EtOH. M.p. 95°.
Acid phthalate : cryst. from Et_2O–ligroin. M.p. 81–83·5°.

Skita, *Ber.*, 1920, **53**, 1800.

2 : 4 : 6-Trimethylcyclohexanol.
M.p. 70·5–71°. B.p. 182–4°.
α-Naphthylurethane : m.p. 197·5–198°.

Ungnade, McLaren, *J. Am. Chem. Soc.*, 1944, **66**, 121.

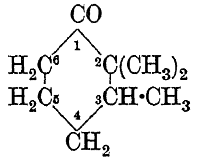
2 : 5 : 5-Trimethylcyclohexanol.
Liq. B.p. 86–9°/18 mm.
Acid phthalate : m.p. 155–155·5°.

Hirsjärvi, *Chem. Abstracts*, 1939, **33**, 6255; 1948, **42**, 2936.

3 : 3 : 5-Trimethylcyclohexanol (*Dihydroisophorol*).
Cis :
B.p. 201–3°/750 mm., 92°/12 mm. D_4^{16} 0·9006. n_D^{16} 1·4550.
Acetyl : b.p. about 209–10°.
Trans :
Two forms. (i) M.p. 37°. B.p. 197·6°. D_{20}^{40} 0·8778. (ii) M.p. 55·8°. B.p. 189·2°. D_{20}^{60} 0·8643. Very slightly sol. H_2O.

Skita, Meyer, *Ber.*, 1912, **45**, 3593.
Knoevenagel, Fischer, *Ann.*, 1897, **297**, 128, 194.

2 : 2 : 3-Trimethylcyclohexanone



$C_9H_{16}O$ MW, 140

Liq. B.p. 66–7°/10 mm. D_4^{15} 0·9234. n_D^{15} 1·4569.
Semicarbazone : cryst. from MeOH. M.p. 203–4°.
2 : 4-*Dinitrophenylhydrazone* : cryst. from EtOH. M.p. 134–6°.

Schäppi, Seidel, *Helv. Chim. Acta*, 1947, **30**, 2206.
Ruzicka *et al.*, *Helv. Chim. Acta*, 1948, **31**, 430.
Horning, Horning, Platt, *J. Am. Chem. Soc.*, 1949, **71**, 1773.

2 : 2 : 4-Trimethylcyclohexanone.
Liq. B.p. 86–7°/12 mm. D_4^{20} 0·8914. n_D^{20} 1·4520.
Oxime : m.p. 62°.
Semicarbazone : m.p. 212° (200–1°).

Godchot, Cauquil, *Compt. rend.*, 1937, **204**, 733.
Calas, *Compt. rend.*, 1939, **208**, 1413.

2 : 2 : 5-Trimethylcyclohexanone.
See Pulenone.

2 : 2 : 6-Trimethylcyclohexanone.
B.p. 178–9°, 66–7°/10 mm. D_4^{18} 0·9043. n_D^{18} 1·4493.
Oxime : cryst. from EtOH. M.p. 106° (104·5–105°). B.p. 126–7°/17 mm.
Semicarbazone : m.p. 220–1°.

Masson, *Compt. rend.*, 1912, **154**, 517.
Haller, Cornubert, *Bull. soc. chim.*, 1927, **41**, 377.
Cornubert, *ibid.*, 894.

2 : 3 : 3-Trimethylcyclohexanone.
Liq. B.p. 190·5–191°/750 mm. D_{15}^{15} 0·9213.
Oxime : needles from pet. ether or EtOH.Aq. M.p. 95°. Sol. most org. solvents.
Semicarbazone : cryst. from EtOH. M.p. 206° decomp. Spar. sol. AcOEt, $CHCl_3$, Me_2CO, C_6H_6.

Crossley, Renouf, *J. Chem. Soc.*, 1911, **99**, 1110.

2 : 3 : 6-Trimethylcyclohexanone.
B.p. 190–1°, 79–80°/20 mm. D_4^{18} 0·9129, D^{21} 0·9058. n_D^{21} 1·4464. Non-misc. with H_2O. Does not form bisulphite comp.

Oxime : m.p. 139·5–140·5°.
Semicarbazone : m.p. 173–4°.

Cornubert, Humeau, *Bull. soc. chim.*, 1931, **49**, 1483.
Zelinsky, Reformatski, *Ber.*, 1895, **28**, 2944.
Ungnade, McLaren, *J. Org. Chem.*, 1945, **10**, 30.

2 : 4 : 4-Trimethylcyclohexanone.
Present in acetone oil. B.p. 191°, 61°/11 mm. D_4^{20} 0·902. n_D^{20} 1·4493.
Oxime : cryst. from Et_2O. M.p. 108–9°.
Semicarbazone : m.p. 164–5° (162°).

Auwers, *Ann.*, 1920, **420**, 111.
Pringsheim, Bondi, *Ber.*, 1925, **58**, 1414.
Wallach, Scheunert, *Ann.*, 1902, **324**, 107.

2 : 4 : 5-Trimethylcyclohexanone.
Cis :
B.p. 193°. D_4^{20} 0·897. n_D^{20} 1·4479.
Oxime : cryst. from MeOH. M.p. 105°.
Semicarbazone : needles from MeOH. M.p. 204° (175·5–176°).

Skita, *Ber.*, 1920, **53**, 1800.
Auwers, *Ann.*, 1920, **420**, 103.

2 : 4 : 6-Trimethylcyclohexanone.
B.p. 184–5°/748 mm. D_4^{20} 0·8992. n_D^{20} 1·4458.
Oxime : m.p. 40–1°. B.p. 125–7°/21 mm.
Semicarbazone : m.p. 228° (216·5–217·5°).

Cornubert, Maurel, *Bull. soc. chim.*, 1931, **49**, 1528.
Haller, *Compt. rend.*, 1913, **157**, 740.
Ungnade, McLaren, *J. Org. Chem.*, 1945, **10**, 30.

2 : 5 : 5-Trimethylcyclohexanone.
B.p. 185°.
Semicarbazone : m.p. 170°.

Blanc, *Bull. soc. chim.*, 1908, **3**, 786.

3 : 3 : 4-Trimethylcyclohexanone.
B.p. 184–8°, 70–5°/11 mm.
Oxime : cryst. from H_2O. M.p. 100°.
Semicarbazone : m.p. 177°.

v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 188.

3 : 3 : 5-Trimethylcyclohexanone (*Dihydroisophorone*).
Present in acetone oil. Yellow oil. B.p. 188·5–189·5°, 53·5–54°/11 mm. D^{19} 0·8919. n_D^{15} 1·4454.
Oxime : cryst. from EtOH. M.p. 58°.
Semicarbazone : cryst. from MeOH. M.p. 204° (202°).
2 : 4-*Dinitrophenylhydrazone* : m.p. 145–7°.

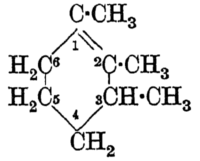
Pringsheim, Bondi, *Ber.*, 1925, **58**, 1414.
Knoevenagel, *Ann.*, 1897, **297**, 198.
Skita, *Ber.*, 1909, **42**, 1630.

3 : 4 : 4-Trimethylcyclohexanone.
Liq. B.p. 80–1°/13 mm. D_4^{25} 0·911. n_D^{20} 1·4552. Forms a bisulphite comp.

Oxime : cryst. from EtOH. M.p. 72·5–73·5°.
Semicarbazone : cryst. from EtOH.Aq. M.p. 208–9°.
*Di-*p*-nitrobenzylidene deriv.* : cryst. from C_6H_6. M.p. 223–223·2°.

Buchman, Sargent, *J. Org. Chem.*, 1942, **7**, 144.

1 : 2 : 3-Trimethylcyclohexene



C_9H_{16} MW, 124

B.p. 149·6–150°/749 mm. $D_4^{11·75}$ 0·8347. $n_D^{11·75}$ 1·463.

Auwers, Krollpfeiffer, *Ber.*, 1915, **48**, 1231.

1 : 3 : 5-Trimethylcyclohexene (*Tetrahydromesitylene*).
B.p. 142·5–143·5°. $D_4^{14·3}$ 0·8025. $n_D^{13·5}$ 1·449.
Nitrosochloride : m.p. 134°.

Wallach, *Ann.*, 1913, **396**, 284.
Auwers, Hinterseber, Treppmann, *Ann.*, 1915, **410**, 270.

1 : 4 : 4-Trimethylcyclohexene.
See Pulenene.

1 : 4 : 5-Trimethylcyclohexene (1 : 2 : 3 : 6-*Tetrahydro-ψ-cumene*).
B.p. 144–6°. D_4^{20} 0·805. n_D^{20} 1·4482.

Auwers, *Ann.*, 1920, **420**, 105.

1 : 5 : 5-Trimethylcyclohexene (*α-Cyclogeraniolene*).
B.p. 139–41°/759 mm. D^{23} 0·7981. $n_D^{21·5}$ 1·44612.
Nitrosate : cryst. M.p. 102–4°.
Nitrosochloride : bluish cryst. from MeOH.Aq. M.p. 100–20°.

Wallach, Scheunert, *Ann.*, 1902, **324**, 101.
Crossley, Gilling, *J. Chem. Soc.*, 1910, **97**, 2221.

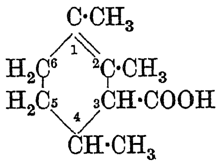
1 : 5 : 6-Trimethylcyclohexene.
Liq. B.p. 140°/760 mm. D_{25}^{25} 0·831. n_D^{25} 1·4572.

Mousseron, Winternitz, Jacquier, *Compt. rend.*, 1947, **224**, 1064.

1 : 6 : 6-Trimethylcyclohexene.
Liq. B.p. 146·2–147·2°/767 mm. (144–6°). $D_4^{20·3}$ 0·8217. $n_D^{20·4}$ 1·456.
Nitrosochloride : needles from AcOEt. M.p. 133–4°. Insol. MeOH, Me_2CO.

Auwers, Lange, *Ann.*, 1915, **409**, 174.
Godchot, Bedos, *Compt. rend.*, 1925, **181**, 921.
Colonge, Duroux, *Bull. soc. chim.*, 1940, **7**, 467.

1 : 2 : 4-Trimethylcyclohexene-3-carboxylic Acid



$C_{10}H_{16}O_2$ MW, 168

Cryst. from AcOH.Aq. M.p. 56°.

Me ester: b.p. 106°/19 mm. D_4^{16} 0·9738. n_D^{16} 1·4679.

Anilide: needles from MeOH. M.p. 94·5°.

Farmer, Pitkethly, *J. Chem. Soc.*, 1938, 17.

1 : 2 : 4-Trimethylcyclohexene-4-carboxylic Acid.

Cryst. from 50% AcOH.Aq. M.p. 55·5–56·5°.

Me ester: b.p. 103–103·5°/20 mm. D_4^{20} 0·9696. n_D^{20} 1·4666.

Petrov, Sopov, *Chem. Abstracts*, 1949, **43**, 3373.

1 : 2 : 4-Trimethylcyclohexene-5-carboxylic Acid.

Colourless leaflets from AcOH.Aq. M.p. 137°.

Et ester: b.p. 127–30°/30 mm. D_0^{20} 0·9594. n_D^{20} 1·4607.

Anilide: needles from MeOH. M.p. 162°.

Farmer, Pitkethly, *J. Chem. Soc.*, 1938, 19.

1 : 3 : 3 - Trimethylcyclohexene-2-carboxylic Acid.

See Δ^1-Cyclogeranic Acid.

1 : 3 : 3-Trimethylcyclohexene-4-carboxylic Acid.

Needles from AcOH.Aq. M.p. 86–7°.

Jitkow, Bogert, *J. Am. Chem. Soc.*, 1941, **63**, 1981.

Penfold, Simonsen, *J. Chem. Soc.*, 1942, 206.

1 : 5 : 5-Trimethylcyclohexene-6-carboxylic Acid.

See Δ^2-Cyclogeranic Acid.

3 : 3 : 4-Trimethylcyclohexene-2-carboxylic Acid.

Plates from MeOH. M.p. 135°.

Chakravarti, *J. Chem. Soc.*, 1947, 1567.

3 : 3 : 4-Trimethylcyclohexene-4-carboxylic Acid.

See Camphorenic Acid.

3 : 3 : 5-Trimethylcyclohexene-4-carboxylic Acid.

See Δ^4-Cyclogeranic Acid.

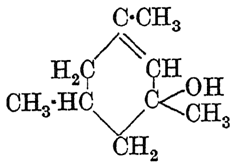
3 : 5 : 5-Trimethylcyclohexene-4-carboxylic Acid.

See Δ^3-Cyclogeranic Acid.

Trimethylcyclohexene-aldehyde.

See Cyclocitral.

1 : 3 : 5-Trimethylcyclohexenol-3



$C_9H_{16}O$ MW, 140

Cryst. M.p. 46°. B.p. 87–90°/17 mm. $D_4^{20\cdot2}$ 0·9132. $n_D^{19\cdot3}$ 1·4735. Heat of comb. C_v 1350–9 Cal.

Auwers, Peters, *Ber.*, 1910, **43**, 3087.

1 : 5 : 5-Trimethylcyclohexenol-3.

Liq. B.p. 69°/5 mm. D_{20}^{20} 0·9144. n_D^{20} 1·4717.

Benzoyl: b.p. 134–6°/5 mm. n_D^{20} 1·5198.

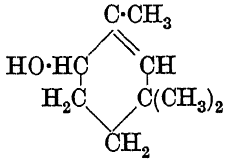
p-*Nitrobenzoyl*: plates from MeOH. M.p. 68·5–69·5°.

Kharasch, Tawney, *J. Am. Chem. Soc.*, 1941, **63**, 2314.

3 : 3 : 6-Trimethylcyclohexenol-4.

See βγ-Pulenenol.

1 : 3 : 3-Trimethylcyclohexenol-6



$C_9H_{16}O$ MW, 140

Oil. B.p. 193°/760 mm. D_4^{13} 0·9310. Misc. with usual org. solvents. Insol. H_2O. Volatile in steam.

Acetyl: b.p. 206–7°.

Bougault, *Compt. rend.*, 1910, **150**, 534.

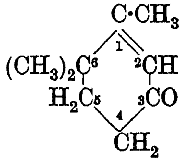
1 : 4 : 4-Trimethylcyclohexenone-3.

See αβ-Pulenenone.

1 : 5 : 5-Trimethylcyclohexenone-3.

See Isophorone.

1 : 6 : 6-Trimethylcyclohexenone-3



$C_9H_{14}O$ MW, 138

Liq. B.p. 217°/760 mm., 97–9°/13 mm. D_4^{25} 0·944. n_D^{25} 1·4889.

Oxime: cryst. from pet. ether. M.p. 53·8–54·3°.

Semicarbazone: needles from AcOEt. M.p. 211° (206·5–207°).

Tiemann, *Ber.*, 1897, **30**, 249.

Buchman, Sargent, *J. Org. Chem.*, 1942, **7**, 144.

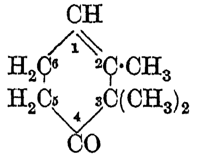
2 : 6 : 6-Trimethylcyclohexenone-3.
B.p. 194–6° (192°). D_4^{20} 0·930. n_D^{20} 1·4779.
Oxime: prisms from Et_2O–ligroin. M.p. 128–9°. B.p. 131–2°/15 mm.
Semicarbazone: cryst. from MeOH.Aq. M.p. 158–9°. Mod. sol. Et_2O.

Bougault, *Compt. rend.*, 1910, **150**, 534.
Auwers, *Ann.*, 1920, **420**, 110.
Wallach, Scheunert, *Ann.*, 1902, **324**, 103.

4 : 4 : 5-Trimethylcyclohexenone-3.
B.p. 85–90°/14 mm.
Semicarbazone: cryst. from EtOH. M.p. 185–7°.

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 159.

2 : 3 : 3-Trimethylcyclohexenone-4



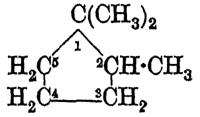
$C_9H_{14}O$ MW, 138

B.p. 85–90°/12 mm.
Semicarbazone: cryst. from MeOH. M.p. 168–71°.

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 158.

3 : 3 : 6-Trimethylcyclohexenone-4.
See βγ-Pulenenone.

1 : 1 : 2-Trimethylcyclopentane (*Camphoceane, dihydroisolaurolene*)



C_8H_{16} MW, 112

Liq. with camphoraceous odour. B.p. 113–113·5°/750 mm. D_4^{18} 0·7728, D_0^{20} 0·7661. n_D^{18} 1·4238, n_D^{20} 1·4199. Heat of comb. C_v 1252·8 Cal. Dil. HNO_3 ⟶ 1 : 1-dimethylglutaric acid.

Zelinsky, Lepeschkin, *Ann.*, 1901, **319**, 315.
Crossley, Renouf, *J. Chem. Soc.*, 1906, **89**, 43.
Kishner, *Chem. Zentr.*, 1911, I, 543.

1 : 1 : 3-Trimethylcyclopentane.
B.p. 105°/760 mm. D_4^{18} 0·7764, D_4^{20} 0·7703 (0·7481). n_D^{18} 1·4240, n_D^{20} 1·4223 (1·4109).

Zelinsky, Uspensky, *Ber.*, 1913, **46**, 1470.
Dey, Linstead, *J. Chem. Soc.*, 1935, 1064.
McKinley, Stevens, Baldwin, *J. Am. Chem. Soc.*, 1945, **67**, 1458.

1 : 2 : 3-Trimethylcyclopentane (*Dihydrolaurolene, laurolan*).
Cis-cis-cis:
B.p. 122·0–122·1°/760 mm. D_4^{20} 0·7766. n_D^{20} 1·4250.
Cis-cis-trans:
B.p. 118–118·2°/760 mm. D_4^{20} 0·7695. n_D^{20} 1·4216.
Cis-trans-cis:
B.p. 110·2–110·3°/760 mm. D_4^{20} 0·7540. n_D^{20} 1·4140.

Zelinsky, Lepeschkin, *Chem. Zentr.*, 1902, I, 33.
Crossley, Renouf, *J. Chem. Soc.*, 1906, **89**, 27, 40.
Koperina, Kazanskiĭ, *Chem. Abstracts*, 1949, **43**, 156.

1 : 2 : 4-Trimethylcyclopentane.
B.p. 112·5–113°. D_4^{20} 0·7565. n_D^{20} 1·4156. Heat of comb. C_v 1255·7 Cal.

Zelinsky, *Bull. soc. chim.*, 1904, **32**, 747.

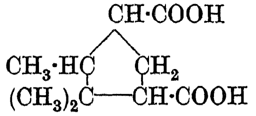
1 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid.
See Laurolanic Acid.

2 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid.
See Norisocampholic Acid.

1 : 1 : 2-Trimethylcyclopentane-5-carboxylic Acid.
See Lauronic Acid.

1 : 2 : 2-Trimethylcyclopentane-1 : 3-dicarboxylic Acid.
See Camphoric Acid.

4 : 4 : 5-Trimethylcyclopentane-1 : 3-dicarboxylic Acid

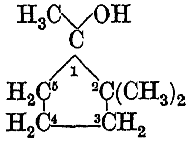


$C_{10}H_{16}O_4$ MW, 200

Cis:
Cryst. from H_2O. M.p. 225–6°.
Trans:
M.p. 189–90°.

Zabrodina, *Chem. Abstracts*, 1949, **43**, 2605.

1 : 2 : 2-Trimethylcyclopentanol



$C_8H_{16}O$ MW, 128

Liq. B.p. 156°/755 mm., 80–1°/49 mm., 60°/15 mm. D_0^{20} 0·9102. n_D^{20} 1·4513. Dist. with cryst. oxalic acid ⟶ isolaurolene.
Hydrate, $\frac{1}{2}H_2O$: needles from Et_2O. M.p. 59–60°.

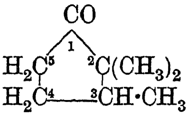
Kishner, *Chem. Zentr.*, 1911, I, 543.

1 : 2 : 4-Trimethylcyclopentanol.
d-.
B.p. 157–8°/747 mm. D_4^{21} 0·8850. n_D^{21} 1·4424. $[\alpha]_D + 15°$.
Zelinsky, *Bull. soc. chim.*, 1904, **32**, 747.

1 : 2 : 5-Trimethylcyclopentanol.
B.p. 56–60°/8 mm. D_4^{15} 0·9121. $n_D^{16\cdot7}$ 1·4554. Heat with anhyd. oxalic acid or $P_2O_5 \longrightarrow$ laurolene.
Noyes, Kyriakides, *J. Am. Chem. Soc.*, 1910, **32**, 1065.

2 : 3 : 4-Trimethylcyclopentanol.
Liq. with menthol odour. B.p. 177·5–178·5°/775 mm., 68–70°/12 mm. D_4^{20} 0·9102. n_D^{20} 1·4653. Insol. H_2O.
Willstätter, Clarke, *Ber.*, 1914, **47**, 309.
Koperina, Kazanskiĭ, *Chem. Abstracts*, 1949, **43**, 156.

2 : 2 : 3-Trimethylcyclopentanone



$C_8H_{14}O$ MW, 126
Exists in active and inactive forms.
Active form :
Liq. B.p. 164–5°.
Oxime : leaflets from pet. ether. M.p. 107–8°. Sol. EtOH, pet. ether.
Semicarbazone : m.p. 188°. Spar. sol. cold EtOH.
Inactive form :
B.p. 164°.
Oxime : plates. M.p. 105°.
Semicarbazone : m.p. 210–12°.
Blaise, Blanc, *Bull. soc. chim.*, 1902, **27**, 76.
Blanc, Desfontaines, *Compt. rend.*, 1903, **136**, 1143.
Schäppi, Seidel, *Helv. Chim. Acta*, 1947, **30**, 2206.
Ruzicka, Seidel, Schinz, Pfeiffer, *Helv. Chim. Acta*, 1948, **31**, 430.

2 : 2 : 4-Trimethylcyclopentanone.
Liq. B.p. 153–4°/742 mm. D_4^{26} 0·871. n_D^{26} 1·4279.
Oxime : cryst. from EtOH. M.p. 79·7–80°.
Semicarbazone : cryst. from EtOH. M.p. 171·0–171·3°.
Sargent, *J. Org. Chem.*, 1942, **7**, 157.

2 : 2 : 5-Trimethylcyclopentanone.
Exists in active and inactive forms.
Active form :
B.p. 152–3°.
Oxime : cryst. from MeOH.Aq. M.p. 60–2°. Very sol. org. solvents.
Semicarbazone : m.p. 150–1°. Very sol. MeOH, Et_2O.
Inactive form :
B.p. 152°. D_4^{20} 0·8781. n_D^{20} 1·4306.
Oxime : prisms from pet. ether. M.p. 62°.
Blanc, *Bull. soc. chim.*, 1908, **3**, 290, 782.
Haller, Cornubert, *Compt. rend.*, 1914, **158**, 300.
Wallach, Kempe, *Ann.*, 1903, **329**, 94.

2 : 3 : 3-Trimethylcyclopentanone.
Liq. B.p. 167–9°/760 mm. D_4^{20} 0·8956. Insol. H_2O.
Oxime : needles from EtOH.Aq. M.p. 105·5°.
Semicarbazone : plates from EtOH. M.p. 221·5–222° decomp.
van Kregten, *Rec. trav. chim.*, 1916, **36**, 77.
Noyes, *Ber.*, 1899, **32**, 2291.

2 : 3 : 5-Trimethylcyclopentanone.
d-.
B.p. 158–9°/770 mm. D_4^{19} 0·8778. n_D^{19} 1·4316. $[\alpha]_D^{19} + 103° 41'$.
Haller, Cornubert, *Compt. rend.*, 1914, **158**, 1619.

3 : 3 : 4-Trimethylcyclopentanone.
Liq. B.p. 172–4°. D_4^{25} 0·892. n_D^{25} 1·4386.
Oxime : cryst. from EtOH. M.p. 99·8—100°.
Semicarbazone : cryst. from EtOH. M.p. 213·5–214°.
v. Braun, *Ann.*, 1931, **490**, 132.
Buchman, Sargent, *J. Org. Chem.*, 1942, **7**, 151.
See also v. Braun, Mannes, Reuter, *Ber.*, 1933, **66**, 1499.

3 : 3 : 5-Trimethylcyclopentanone.
B.p. 160–1°. D_4^{18} 0·8785. n_D^{18} 1·433.
Semicarbazone : cryst. from EtOH. M.p. 171–3°.
Dey, Linstead, *J. Chem. Soc.*, 1935, 1063.
Wallach, *Ann.*, 1918, **414**, 331.

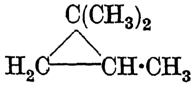
1 : 2 : 3-Trimethylcyclopentene.
See Laurolene.

1 : 5 : 5-Trimethylcyclopentene.
See Isolaurolene.

Trimethylcyclopentene-carboxylic Acid.
See Campholytic Acid *and* Laurolenic Acid.

2 : 2 : 3-Trimethylcyclopentylacetic Acid.
See Isocampholic Acid.

1 : 1 : 2-Trimethylcyclopropane



C_6H_{12} MW, 84
B.p. 56–7°/750 mm. (52·8°/756 mm.). D_4^{15} 0·6888, $D_4^{19\cdot5}$ 0·6822. $n_D^{14\cdot5}$ 1·3896, $n_D^{19\cdot5}$ 1·3848. Polymerises on shaking with conc. H_2SO_4.
Kishner, *Chem. Zentr.*, 1912, I, 2025.
Zelinsky, Zelikow, *Ber.*, 1901, **34**, 2859.
Kelso *et al.*, *J. Am. Chem. Soc.*, 1952, **74**, 287.

1 : 2 : 3-Trimethylcyclopropane.
B.p. 65–7°/755 mm. D_4^{18} 0·6946. n_D^{18} 1·3945.

Zelinsky, Zelikow, *Ber.*, 1901, **34**, 2863.

1 : 3 : 5-Trimethyldiphenylmethane.
See Phenylmesitylmethane.

Trimethyldiphenyl sulphide.
See under Thioxylenol.

2 : 6 : 10-Trimethyldodecane.
See Farnesane.

11 : 15 : 19-Trimethyleicosa-1 : 11-diene

$$\begin{array}{l} \quad\;\; CH_3 \qquad\qquad\quad CH_3 \qquad\qquad\qquad CH_3 \\ CH_3{\cdot}CH{\cdot}[CH_2]_3{\cdot}CH{\cdot}[CH_2]_2{\cdot}CH{:}C{\cdot}[CH_2]_8{\cdot}CH{:}CH_2 \end{array}$$

$C_{23}H_{44}$ MW, 320

Mobile liq. B.p. 162–3°/0·3 mm. n_D^{19} 1·4595. Misc. with C_6H_6.

Polgar, Robinson, *J. Chem. Soc.*, 1945, 395.

Trimethylene.
See Cyclopropane.

Trimethylene-aniline.
See under Trimethyleneimine.

Trimethylene bromide.
See 1 : 3-Dibromopropane.

Trimethylene bromohydrin.
See 3-Bromopropyl Alcohol.

Trimethylene chloride.
See 1 : 3-Dichloropropane.

Trimethylene chlorobromide.
See 1-Chloro-3-bromopropane.

Trimethylene chlorohydrin.
See 3-Chloropropyl Alcohol.

Trimethylene chloroiodide.
See 3-Chloro-1-iodopropane.

Trimethylene cyanide.
See under Glutaric Acid.

Trimethylenediamine (1 : 3-*Diaminopropane*)

$$H_2N{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2$$

$C_3H_{10}N_2$ MW, 74

Liq. with ammoniacal odour. B.p. 135–6°/738 mm. D_4^{25} 0·884. n_D^{20} 1·4600. Misc. with EtOH, Et_2O, $CHCl_3$, C_6H_6. $k = 3{\cdot}5 \times 10^{-4}$ at 25°.

B,2HCl: cryst. M.p. 243°. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O.

B,H_2PtCl_6: m.p. 240° decomp.

1 : 3-N-*Tetra-Me*: $C_7H_{18}N_2$. MW, 130. Oil. B.p. 144°/760 mm. $D_4^{18.7}$ 0·7837. $n_\alpha^{18.7}$ 1·4215. Misc. with H_2O, EtOH, Et_2O. *B,H_2PtCl_6*: cryst. from 50% EtOH. Decomp. at 246–7°. *Picrate*: cryst. M.p. 205°.

1-N-*Di-Et*: $C_7H_{18}N_2$. MW, 130. B.p. 75°/20 mm.

1 : 3-N-*Tetra-Et*: $C_{11}H_{26}N_2$. MW, 186. B.p. 205–9°. *$B,2HCl,HgCl_2$*: prisms from H_2O. M.p. 124–5°.

1 : 3-N-*Diacetyl*: prisms from EtOH. M.p. 101°. Sol. H_2O, EtOH, $CHCl_3$. Spar. sol. C_6H_6. Insol. Et_2O, ligroin. Weak base.

1 : 3-N-*Dibenzoyl*: cryst. powder from C_6H_6. M.p. 147–8°. Sol. EtOH, $CHCl_3$. Mod. sol. cold C_6H_6.

1 : 3-N-*Dicarbomethoxyl*: plates from Et_2O. M.p. 74–5°. Sol. H_2O, EtOH, $CHCl_3$. Spar. sol. Et_2O. Insol. C_6H_6, ligroin.

1 : 3-N-*Dicarbethoxyl*: prisms from Et_2O. M.p. 42°. B.p. 210°/30 mm. Sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. ligroin. Insol. H_2O.

Dipicrate: yellow leaflets from H_2O. M.p. 250°.

Clarke, *J. Chem. Soc.*, 1913, **103**, 1699.
Fischer, Koch, *Ber.*, 1884, **17**, 1799.
Putochin, *Ber.*, 1926, **59**, 625.
Ing, Manske, *J. Chem. Soc.*, 1926, 2351.
Wiedeman, Montgomery, *J. Am. Chem. Soc.*, 1945, **67**, 1996.
Whitmore *et al.*, *J. Am. Chem. Soc.*, 1944, **66**, 728.
Aspinall, *J. Am. Chem. Soc.*, 1941, **63**, 2843.

Trimethylene Dithioglycol (1 : 3-*Dimercaptopropane, dithiotrimethylene glycol, propane-1 : 3-dithiol*)

$$HS{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}SH$$

$C_3H_8S_2$ MW, 108

Oil. B.p. 94°/60 mm., 64·5°/15 mm. D_4^{20} 1·08.

Simpson, *Can. J. Research*, 1947, **25*B***, 20.

Trimethylene Glycol (1 : 3-*Dihydroxypropane, propandiol*-1 : 3)

$$HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}OH$$

$C_3H_8O_2$ MW, 76

Pale yellow liq. B.p. 210–11°, 109–10°/12 mm. D_4^{20} 1·0597. n_D^{20} 1·43983. Misc. with H_2O, EtOH. $H_2O_2 \longrightarrow$ propionaldehyde.

$B,2HNO_3$: viscous liq. D^{15} 1·408. Sol. MeOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Sol. 5 parts EtOH, 410 parts H_2O. Spar. sol. CS_2.

Me ether: $C_4H_{10}O_2$. MW, 90. B.p. 153·15–153·2°/768 mm. D_4^{20} 0·9434. n_D^{20} 1·41259. *Phenyl ether*: b.p. 230–1°. *Formyl*: b.p. 146–7°/767 mm. D_4^{15} 1·0057. *Acetyl*: b.p. 162–163·5°/762 mm. D_4^{15} 0·9803.

Et ether: $C_5H_{12}O_2$. MW, 104. B.p. 162·1–162·2°. D_4^{20} 0·91691. n_D^{20} 1·41666. Misc. with H_2O. *Phenyl ether*: b.p. 328–30°. *Formyl*: b.p. 157·5–159°/742 mm. D_4^{15} 0·9731. *Acetyl*: b.p. 174·5–175·5°. D_4^{15} 0·9567.

Di-Et ether: $C_7H_{16}O_2$. MW, 132. B.p. 140–1°. D_{25}^{25} 0·835. Insol. H_2O.

Propyl ether: $C_6H_{14}O_2$. MW, 118. B.p. 170–2°. D_4^{15} 0·9076. *Formyl*: b.p. 174·5–176°.

Methylene ether: *see* 1 : 3-Dioxan.

Phenyl ether: $C_9H_{12}O_2$. MW, 152. B.p. 249–50°/764 mm.

Diphenyl ether: 1 : 3-diphenoxypropane. $C_{15}H_{16}O_2$. MW, 228. Leaflets from EtOH.

M.p. 61°. B.p. 338–40°. Sol. EtOH, Et_2O. Insol. H_2O.

Di-o-tolyl ether: b.p. 341–3°, 225°/28 mm.

Di-m-tolyl ether: plates from EtOH. M.p. 91°.

Di-p-tolyl ether: needles from EtOH. M.p. 94°.

Di-1-naphthyl ether: needles from EtOH. M.p. 103–4°.

Di-2-naphthyl ether: plates from AcOH. M.p. 148–9°.

Formyl: b.p. 195–7°/757 mm. D_4^{15} 1·1405.

Acetyl: b.p. 202·5–204°.

Diacetyl: b.p. 209–10°. D^{19} 1·070. Sol. 8–10 parts H_2O.

Dibenzoyl: prisms or needles from pet. ether. M.p. 57·5° (53°).

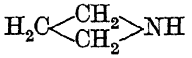
Di-p-toluenesulphonyl: m.p. 93–4°.

Diphenylurethane: m.p. 137–137·5°.

Di-1-naphthylurethane: m.p. 164°.

Gattermann, *Ann.*, 1907, **357**, 379.
Henry, *Chem. Zentr.*, 1899, I, 968; 1907, I, 1314.
Blechta, *Chem. Abstracts*, 1922, **16**, 2991.
Rayner, *J. Soc. Chem. Ind.*, 1926, **45**, 265T, 287T.
Werkmann, Gillen, *J. Bact.*, 1932, **23**, 167.
Palomaa, *Chem. Zentr.*, 1913, II, 1959.

Trimethyleneimine (*Azetidine*)



C_3H_7N MW, 57

Liq. with ammoniacal odour. B.p. 63°/748 mm. $D^{20\cdot4}$ 0·8436. n_D^{24} 1·4287. Misc. with H_2O, EtOH.

B,HAuCl₄: golden-yellow cryst. M.p. 192°. Spar. sol. cold H_2O.

B_2,H_2PtCl_6: orange-yellow needles. M.p. 203° decomp.

Picrate: yellow needles. M.p. 166–7°.

N-*Phenyl*: trimethylene-aniline. Oil. B.p. 242–5°, 130–2°/16 mm.

Howard, Marckwald, *Ber.*, 1899, **32**, 2032.
Scholtz, *ibid.*, 2255.
Ruzicka, Salomon, Meyer, *Helv. Chim. Acta*, 1937, **20**, 126.
Yanbikov, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 1470, 1545.
Jones, *J. Org. Chem.*, 1944, **9**, 491.

Trimethylene iodide.
See 1 : 3-Di-iodopropane.

Trimethylene iodohydrin.
See 3-Iodopropyl Alcohol.

Trimethylene methylene dioxide.
See 1 : 3-Dioxan.

Trimethylene oxide

C_3H_6O MW, 58

Liq. with pleasant aromatic odour. B.p. 47·8°. D_4^{25} 0·8930. n^{25} 1·3897. Misc. with H_2O.

Allen, Hibbert, *J. Am. Chem. Soc.*, 1934, **56**, 1399.
Derick, Bissell, *J. Am. Chem. Soc.*, 1916, **38**, 2478.

2 : 6-Trimethylenepiperidine.
See Granatanine.

2 : 5-Trimethylenepyrrolidine.
See Nortropane.

Trimethylene sulphide

C_3H_6S MW, 74

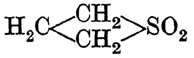
Mobile liq. with disagreeable odour. B.p. 93·8–94·2°/752 mm. D_4^{23} 1·0284. n_D^{23} 1·5059.

Dimethiodide: needles. M.p. 98·5–99·5°.

$B,HgCl_2$: cryst. Decomp. at 93–5°. Spar. sol. H_2O, EtOH, Et_2O.

Bost, Conn, *Oil and Gas Journal*, 1933, **32**, No. 3, 17.
Grischkewitsch-Trochimowski, *Chem. Zentr.*, 1923, III, 773.

Trimethylene sulphone



$C_3H_6O_2S$ MW, 106

Needles from H_2O, MeOH–Et_2O, or Et_2O–pet. ether. M.p. 75·5–76°. Sol. H_2O, EtOH. Spar. sol. Et_2O.

Grischkewitsch-Trochimowski, *Chem. Zentr.*, 1923, III, 773

Trimethylene-trinitroamine.
See Cyclonite.

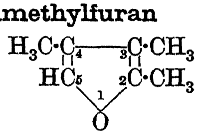
Trimethylethylene.
See 2-Methyl-2-butylene.

Trimethylethylene chlorohydrin.
See 3-Chloro-*tert.*-amyl Alcohol.

1 : 1 : 2 - Trimethyl - 2 - ethylhydracrylic Acid.
See 2-Hydroxy-1 : 1 : 2-trimethyl-*n*-valeric Acid.

2 : 3 : 5-Trimethyl-4-ethylpyrrole.
See Phyllopyrrole.

2 : 3 : 4-Trimethylfuran



$C_7H_{10}O$ MW, 110

Liq. B.p. 54–5°/57 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, **15**, 1116.

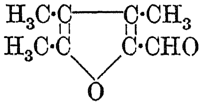
2 : 3 : 5-Trimethylfuran (2 : 4 : 5-*Trimethylfuran*).

B.p. 114°/720 mm., 51·5°/62 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, **15**, 1115.

Trimethylfuran-carboxylic Acid.
See Trimethyl-β-furoic Acid *and* Trimethylpyromucic Acid.

3 : 4 : 5-Trimethylfurfural

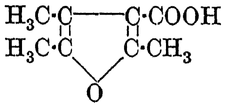


$C_8H_{10}O_2$ MW, 138

Cryst. M.p. 31–2°. B.p. 68°/0·3 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, **15**, 1117.

2 : 4 : 5-Trimethyl-β-furoic Acid (2 : 4 : 5-*Trimethylfuran*-3-*carboxylic acid*)



$C_8H_{10}O_3$ MW, 154

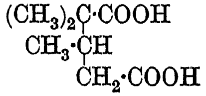
Cryst. from pet. ether. M.p. 131–2°. Sol. most org. solvents except pet. ether. Spar. sol. H_2O.

Et ester: $C_{10}H_{14}O_3$. MW, 182. B.p. 100–5°/12 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, **15**, 1114.

Trimethylglucose.
See under Glucose.

1 : 1 : 2-Trimethylglutaric Acid (1 : 1 : 2-*Trimethylpropane*-1 : 3-*dicarboxylic acid*, 2 : 3-*dimethylbutane*-1 : 3-*dicarboxylic acid*)



$C_8H_{14}O_4$ MW, 174

Cryst. M.p. 112°. Sol. H_2O, most org. solvents.

Di-Et ester: b.p. 128–31°/10 mm.

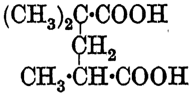
Anhydride: prisms from pet. ether. M.p. 39°.

Monoanilide: plates from EtOH.Aq. M.p. 155°. Sol. most org. solvents.

Mono-p-*toluidide*: cryst. from EtOH.Aq. M.p. 162–3°.

Perkin, Thorpe, *J. Chem. Soc.*, 1897, **71**, 1187.
Ruzicka *et al.*, *Helv. Chim. Acta*, 1942, **25**, 198.

1 : 1 : 3-Trimethylglutaric Acid (2-*Methylpentane*-2 : 4-*dicarboxylic acid*)



$C_8H_{14}O_4$ MW, 174

Leaflets from H_2O. M.p. 97° (95°). Sol. usual org. solvents. Less sol. H_2O. $k = 3{\cdot}48 \times 10^{-5}$ at 25°. Sublimes.

Di-Et ester: $C_{12}H_{22}O_4$. MW, 230. B.p. 230–1°. D^0 1·012.

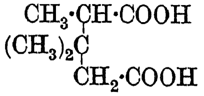
Anhydride: needles from ligroin. M.p. 95–6°. B.p. 262°.

Imide: cryst. M.p. 139°. Sublimes.

Monoanilide: needles. M.p. 165°. Sol. EtOH. Spar. sol. Et_2O. Insol. C_6H_6, ligroin.

Auwers, Meyer, *Ber.*, 1889, 22, 2013.
Auwers, *Ann.*, 1896, **292**, 224; *Ber.*, 1898, **31**, 2113.

1 : 2 : 2-Trimethylglutaric Acid (2 : 2-*Dimethylbutane*-1 : 3-*dicarboxylic acid*)



$C_8H_{14}O_4$ MW, 174

Cryst. from C_6H_6. M.p. 88–9°. Very sol. warm H_2O. Spar. sol. C_6H_6. $k = 1{\cdot}43 \times 10^{-4}$.

Di-Et ester: b.p. 247–9°.

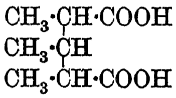
Anhydride: prisms from AcOEt–pet. ether. M.p. 87–8° (82°).

Imide: needles from H_2O. M.p. 126°. Sol. hot H_2O.

Monoanilide: needles from MeOH.Aq. M.p. 150–1°.

Perkin, Thorpe, *J. Chem. Soc.*, 1899, **75**, 65.
Crossley, Renouf, *J. Chem. Soc.*, 1911, **99**, 1108.
Ray, *J. Am. Chem. Soc.*, 1929, **51**, 930.

1 : 2 : 3-Trimethylglutaric Acid (3-*Methylpentane*-2 : 4-*dicarboxylic acid*)

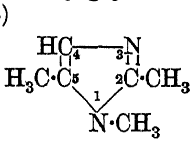


$C_8H_{14}O_4$ MW, 174

Cryst. from C_6H_6. M.p. 134°.

Ray, *J. Am. Chem. Soc.*, 1928, **50**, 558; 1931, **53**, 1174.
See also Michael, Ross, *J. Am. Chem. Soc.*, 1931, **53**, 1175.

1 : 2 : 5-Trimethylglyoxaline (1 : 2 : 5-*Trimethyliminazole*)



$C_6H_{10}N_2$ MW, 110

Brown mobile oil.

B,HAuCl$_4$: orange-yellow plates from dil. HCl. M.p. 186–7°.

Picrate: pale yellow needles from H_2O. M.p. 208–9°.

Grindley, Pyman, *J. Chem. Soc.*, 1927, 3134.

1 : 4 : 5-Trimethylglyoxaline (1 : 4 : 5-*Trimethyliminazole*).

Cryst. M.p. 46°. B.p. 117°/20 mm. Sol. H_2O, EtOH, Et_2O.

B,HCl: cryst. + H_2O. M.p. 80°, anhyd. 199°. Sol. H_2O, EtOH. Insol. Et_2O.

B,HNO₃: cryst. + H_2O. M.p. 46°. Sol. H_2O, EtOH. Insol. Et_2O.

B,HAuCl₄: yellow cryst. M.p. 202°.

B₂,H₂PtCl₆: yellow cryst. from H_2O. M.p. 224–5°.

Picrate: yellow cryst. from H_2O. M.p. 218°.

Methiodide: needles from EtOH–Et_2O. M.p. 158°. Sol. H_2O, EtOH. Insol. Et_2O.

Jowett, *J. Chem. Soc.*, 1905, **87**, 405.

2 : 4 : 5-Trimethylglyoxaline (2 : 4 : 5-*Trimethyliminazole*).

Needles from Et_2O–ligroin. M.p. 132·5–133° (130–1°). Sol. H_2O, most org. solvents. Aq. sol. reacts strongly alkaline. Bitter taste.

B,HCl: needles from EtOH. M.p. 316°.

Picrate: prisms from H_2O. M.p. 163°.

v. Pechmann, *Ber.*, 1888, **21**, 1415.
Fargher, Pyman, *J. Chem. Soc.*, 1919, **115**, 232.

2 : 4 : 6-Trimethylheptanol-4.

See Methyldi-isobutylcarbinol.

Trimethylhexanol.

See Trimethylhexyl Alcohol *and* Isopropyl-*tert.*-amylcarbinol.

3 : 5 : 5-Trimethylhexyl Alcohol (3 : 5 : 5-*Trimethylhexanol*)

$$CH_3\cdot C(CH_3)_2\cdot CH_2\cdot CH(CH_3)\cdot CH_2\cdot CH_2OH$$

$C_9H_{20}O$ MW, 144

B.p. 193–4°/760 mm., 142°/150 mm. D_4^{25} 0·8236. n_D^{25} 1·4300.

Formyl: b.p. 195°/760 mm., 73°/10 mm. n_D^{20} 1·4231.

Acetyl: b.p. 209°/760 mm. n_D^{25} 1·4200.

3 : 5-*Dinitrobenzoyl*: m.p. 62°.

Bruner, *Ind. Eng. Chem.*, 1949, **41**, 2860.
Catlin, Howk, U.S.P. 2,499,848, (*Chem. Abstracts*, 1950, **44**, 5091).
Reid, U.S.P. 2,517,350, (*Chem. Abstracts*, 1950, **44**, 11,173).

Trimethylhydracrylic Acid.

See Hydroxydimethylbutyric Acid.

Trimethylhydroquinone.

See ψ-Cumohydroquinone.

Trimethyl-γ-hydroxy-α-butenyl-cyclohexene.

See Ionol.

Trimethyl-hydroxyethyl-ammonium hydroxide.

See Choline.

Trimethyl-hydroxyisopropyl-ammonium hydroxide.

See α-Methylcholine.

Trimethyl-hydroxymethyl-ammonium hydroxide.

See Formocholine.

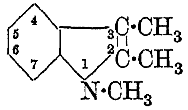
Trimethyl-hydroxypropyl-ammonium hydroxide.

See β-Methylcholine.

Trimethyliminazole.

See Trimethylglyoxaline.

1 : 2 : 3-Trimethylindole



$C_{11}H_{13}N$ MW, 159

Leaflets. M.p. 18°. B.p. 283–4°/750 mm. Very sol. EtOH, Et_2O, C_6H_6. Mod. sol. hot H_2O. Sol. conc. HCl.

Picrate: dark red needles from C_6H_6. M.p. 150°.

Degen, *Ann.*, 1886, **236**, 160.
Ciamician, Piccinini, *Ber.*, 1896, **29**, 2470.
Janetzky, Verkade, *Rec. trav. chim.*, 1946, **65**, 691.

1 : 2 : 5-Trimethylindole.

Leaflets from EtOH.Aq. M.p. 56–7°. Very sol. ligroin. Volatile in steam.

Bayer, D.R.P., 128,660, (*Chem. Zentr.*, 1902, I, 610); 137,117, (*Chem. Zentr.*, 1903, I, 109).

2 : 3 : 4(or 2 : 3 : 6)-Trimethylindole.

Cryst. from EtOH.Aq. M.p. 117–18°.

Picrate: dark red needles from 96% EtOH. M.p. 151–2°.

Janetzky, Verkade, *Rec. trav. chim.*, 1946, **65**, 700.

2 : 3 : 5-Trimethylindole.

Leaflets from EtOH. M.p. 121·5°. B.p. 297°. Sol. EtOH, $CHCl_3$, ligroin. Spar. sol. H_2O. Sol. conc. HCl. Volatile in steam. $FeCl_3$ in AcOH ⟶ green ⟶ blue col.

Picrate: brownish-red needles from EtOH. M.p. 179–80°.

N-*Nitroso*: golden-yellow needles from EtOH. M.p. 73°. Very sol. EtOH, AcOH. Spar. sol. H_2O.

Wolff, *Ber.*, 1888, **21**, 3361.
Grgin, *Monatsh.*, 1906, **27**, 739.
Janetzky, Verkade, *Rec. trav. chim.*, 1946, **65**, 700.

2 : 3 : 7-Trimethylindole.

Leaflets from EtOH. M.p. 79°. B.p. 282–3°.

Picrate: purplish-red needles from C_6H_6–ligroin. M.p. 152°.

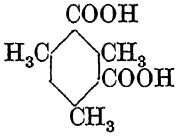
Wolff, *Ber.*, 1888, **21**, 3362.
Janetzky, Verkade, *Rec. trav. chim.*, 1946, **65**, 700.

2 : 4 : 7-Trimethylindole.
Liq. B.p. 158–9°/13 mm.
Picrate: reddish-brown needles from EtOH.Aq. M.p. 175–6°.

v. Braun, Bayer, Blessing, *Ber.*, 1924, **57**, 402.
Allen, Young, Gilbert, *J. Org. Chem.*, 1937, 2, 242.

7 : 8 : 10-Trimethylisoalloxazine.
See Lumilactoflavin.

2 : 4 : 6-Trimethylisophthalic Acid (2 : 4 : 6-*Trimethylbenzene*-1 : 3-*dicarboxylic acid, mesitylene*-2 : 6-*dicarboxylic acid*)



$C_{11}H_{12}O_4$ MW, 208

Cryst. from AcOEt. M.p. 289° (283°). Sol. usual org. solvents.
Dinitrile: $C_{11}H_{10}N_2$. MW, 170. Prisms from EtOH.Aq. M.p. 142°.

Johnson, Fuson, *J. Am. Chem. Soc.*, 1934, **56**, 1418.
Küster, Stallberg, *Ann.*, 1894, **278**, 219.

1 : 3 : 9-Trimethylisoxanthine.
See Isocaffeine.

Trimethylketol (*Dimethylacetylcarbinol*, 2-*methyl*-2-*butanolone*-3, 3-*keto*-tert.-*amyl alcohol*)

$(CH_3)_2C(OH){\cdot}CO{\cdot}CH_3$

$C_5H_{10}O_2$ MW, 102

Liq. with sweet odour. B.p. 141–2°, 50°/18 mm. D_4^{21} 0·9632.
Me ether: *oxime*, prisms from ligroin. M.p. 92–3°. B.p. 190°/742 mm. Sol. H_2O, EtOH. Less sol. ligroin. Volatile in steam.
Oxime: leaflets from ligroin. M.p. 85–6° (96–100°).
Semicarbazone: cryst. from $CHCl_3$–pet. ether. M.p. 165°.
Phenylhydrazone: plates from EtOH. M.p. 83–5°.

Faworsky, *J. prakt. Chem.*, 1913, **88**, 662.
Diels, Johlin, *Ber.*, 1911, **44**, 405.
Schmidt, Austin, *Ber.*, 1902, **35**, 3724.

Trimethyl-lactic Acid.
See Hydroxydimethylbutyric Acid.

Trimethylmalic Acid (*Hydroxytrimethylsuccinic acid*)

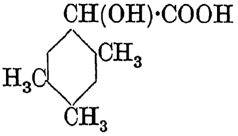
$CH_3{\cdot}C(OH){\cdot}COOH$
$(CH_3)_2C{\cdot}COOH$

$C_7H_{12}O_5$ MW, 176

Plates from H_2O, prisms from AcOEt. M.p. 159–60° (153–5°). M.p. depends on rate of heating. Very sol. H_2O, EtOH, Et_2O, Me_2CO. Sol. AcOEt. Insol. C_6H_6, ligroin. $k = 9{\cdot}4 \times 10^{-4}$ at 25°.
Di-Et ester: $C_{11}H_{20}O_5$. MW, 232. B.p. 122–3°/9 mm. D_{18}^{18} 1·066.
Anhydride: *acetyl*, needles from boiling ligroin. M.p. 67–8°. Sol. usual solvents except H_2O, ligroin.
β-*Lactone*: needles from C_6H_6–pet. ether. M.p. 120° (118–20°).

Auwers, v. Campenhausen, *Ber.*, 1896, **29**, 1544.
Bergroth, Komppa, *Ber.*, 1896, **29**, 1620.
Komppa, *Chem. Zentr.*, 1898, II, 1168; *Ber.*, 1902, **35**, 534.

2 : 4 : 5-Trimethylmandelic Acid (2 : 4 : 5-*Trimethylphenylglycollic acid*)



$C_{11}H_{14}O_3$ MW, 194

Cryst. from C_6H_6. M.p. 137·5–138·5° (133–5°).

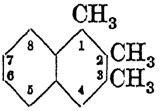
Gattermann, *Ann.*, 1906, **347**, 376.
Fisher, Walling, *J. Am. Chem. Soc.*, 1935, **57**, 1564.
Smith, MacMullen, *J. Am. Chem. Soc.*, 1936, **58**, 633.

2 : 4 : 6-Trimethylmandelic Acid.
See Mesitylglycollic Acid.

Trimethylmethane.
See Isobutane.

Trimethylmethane-tricarboxylic Acid.
See Methane-triacetic Acid.

1 : 2 : 3-Trimethylnaphthalene



$C_{13}H_{14}$ MW, 170

B.p. 125–30°/12 mm.
Picrate: light orange-yellow needles from EtOH. M.p. 142·5°.
Styphnate: yellow. M.p. 143·5°.

Ruzicka, Ehmann, Keller, Schütze, *Helv. Chim. Acta*, 1932, **15**, 143.

1 : 2 : 4-Trimethylnaphthalene.
Leaflets from EtOH. M.p. 50°. B.p. 146°/12 mm.
Picrate: orange needles from MeOH. M.p. 147·5°.
Styphnate: orange-yellow needles. M.p. 123·5°.

Ruzicka, Ehmann, Tombe, Ramondt, *Helv. Chim. Acta*, 1932, **15**, 145.
Hewett, *J. Chem. Soc.*, 1940, 299.

1 : 2 : 5-Trimethylnaphthalene.
M.p. 31–2°. B.p. 147–8°/11 mm. D_4^{22} 1·0103. n_D^{22} 1·6093.
Picrate: cryst. from EtOH. M.p. 137–8°.

Styphnate: cryst. from EtOH. M.p. 131°.
sym.-*Trinitrobenzene add. comp.*: m.p. 159–60°.

Ruzicka, Hosking, *Helv. Chim. Acta*, 1930, **13**, 1411.
Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2546.

1 : 2 : 6-Trimethylnaphthalene.
B.p. 146°/10 mm.
Picrate: orange needles. M.p. 121–2° (120–1°).
Styphnate: yellow needles. M.p. 148°.

Ruzicka, Ehmann, Cuenat, Biasotti, *Helv. Chim. Acta*, 1932, **15**, 146.
Bogert, Apfelbaum, *J. Am. Chem. Soc.*, 1938, **60**, 932.

1 : 2 : 7-Trimethylnaphthalene.
See Sapotalin.

1 : 2 : 8-Trimethylnaphthalene.
B.p. 152–5°/14 mm.
Picrate: reddish-orange needles from EtOH. M.p. 139–40° (133°).
Styphnate: golden-yellow needles from EtOH. M.p. 144·5°.

Ruzicka, Ehmann, Hartnagel, Hausschild, *Helv. Chim. Acta*, 1932, **15**, 149.
Ruzicka, Hofmann, Frei, *Helv. Chim. Acta*, 1936, **19**, 391.

1 : 3 : 5-Trimethylnaphthalene.
Prisms from MeOH. M.p. 47° (43°). B.p. 139·5°/10 mm.
Picrate: orange-yellow needles from MeOH. M.p. 141–2° (140°).
Styphnate: golden-yellow needles. M.p. 138° (136·5°).

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2540.
Ruzicka, Ehmann, Weber, *Helv. Chim. Acta*, 1932, **15**, 153.

1 : 3 : 6-Trimethylnaphthalene.
B.p. 140–4°/10 mm.
Picrate: light orange needles. M.p. 115°.
Styphnate: yellow needles. M.p. 148°.

Ruzicka, Ehmann, Arni, Bernasconi, *Helv. Chim. Acta*, 1932, **15**, 154.

1 : 3 : 7-Trimethylnaphthalene.
B.p. 131–3°/9 mm. D_4^{21} 0·9801. n_D^{15} 1·5972.
Picrate: orange-yellow needles from MeOH. M.p. 142°.
Styphnate: golden-orange cryst. from MeOH. M.p. 151·5°.

Ruzicka, Ehmann, Pieth, Thomann, *Helv. Chim. Acta*, 1932, **15**, 155.

1 : 3 : 8-Trimethylnaphthalene.
Plates from MeOH. M.p. 48°.
Picrate: orange-red needles from EtOH. M.p. 125°.
Styphnate: golden needles from EtOH. M.p. 140·5°.

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2542.

1 : 4 : 5-Trimethylnaphthalene.
Leaflets from MeOH. M.p. 63°. B.p. 145°/12 mm.
Picrate: deep reddish-orange needles. M.p. 144–5°.
Styphnate: reddish-brown needles. M.p. 129–30°.

Ruzicka, Ehmann, Hefti, Altuna, *Helv. Chim. Acta*, 1932, **15**, 156.

1 : 4 : 6-Trimethylnaphthalene.
B.p. 140–2°/15 mm.
Picrate: orange-red needles. M.p. 133°.
Styphnate: golden-yellow cryst. M.p. 114°.

Ruzicka, Ehmann, Addink, *Helv. Chim. Acta*, 1932, **15**, 158.
Buu-Hoï, Cagniant, *Chem. Abstracts*, 1946, **40**, 6070.

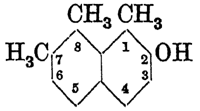
1 : 6 : 7-Trimethylnaphthalene (2 : 3 : 5-*Trimethylnaphthalene*).
Cryst. from MeOH. M.p. 28°. B.p. 138°/12 mm.
Picrate: golden-orange needles from MeOH. M.p. 125° (122·5°).
Styphnate: golden-yellow needles. M.p. 148–9° (146°).

Wilkinson, *J. Chem. Soc.*, 1931, 1333.
Ruzicka, Ehmann, Weisz, *Helv. Chim. Acta*, 1932, **15**, 159.
Barnett, Sanders, *J. Chem. Soc.*, 1933, 437.
Ruzicka, *Helv. Chim. Acta*, 1936, **19**, 423.

2 : 3 : 6-Trimethylnaphthalene.
M.p. 92–3°. B.p. 263–4°, 146–8°/14 mm.
Picrate: orange-yellow needles from MeOH. M.p. 130°.
Styphnate: yellow needles from MeOH. M.p. 165°.

Ruzicka, Ehmann, Rierink, *Helv. Chim. Acta*, 1932, **15**, 160.
Collie, *J. Chem. Soc.*, 1893, **63**, 336.

1 : 7 : 8-Trimethyl-2-naphthol



$C_{13}H_{14}O$ MW, 186

Me ether: cryst. from MeOH. M.p. 74–5°.
sym.-*Trinitrobenzene add. comp.*: cryst. from MeOH. M.p. 128–9°.

Ruzicka, Hofmann, Schellenberg, *Helv. Chim. Acta*, 1936, **19**, 1391.

3 : 4 : 8-Trimethyl-2-naphthol.
Purified by sublimation. M.p. 140–1° decomp.
Me ether: cryst. from EtOH. M.p. 106–7°.

Picrate : cryst. from EtOH. M.p. 150–151·5° decomp.

Picrate : cryst. from EtOH. Very unstable.

Ruzicka, Rey, Smith, *Helv. Chim. Acta*, 1943, **26**, 2057.

3 : 5 : 7-Trimethyl-2-naphthol.

Needles. M.p. 95–6°.

Kruber, *Ber.*, 1939, **72**, 1972.

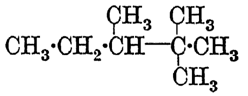
2 : 5 : 8-Trimethylnonanol-5.

See Methyldi-isoamylcarbinol.

3 : 5 : 5-Trimethyloxazolidine-2 : 4-dione.

See Tridione.

2 : 2 : 3-Trimethylpentane



C_8H_{18} MW, 114

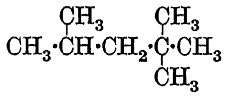
B.p. 109·84°/760 mm. D^{20} 0·71617. n_D^{20} 1·40285.

Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, **55**, 2608.

Brooks, Howard, Crafton, *Journal of Research of the National Bureau of Standards*, 1939, **23**, 637, (*Chem. Abstracts*, 1940, **34**, 1969).

Wibaut *et al.*, *Rec. trav. chim.*, 1939, **58**, 329.

2 : 2 : 4-Trimethylpentane



C_8H_{18} MW, 114

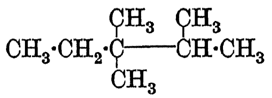
F.p. —107·41°. B.p. 99·3°. D_4^{20} 0·6918. n_D^{20} 1·3916.

Petrov, Andreev, Chapluigin, *Chem. Abstracts*, 1933, **27**, 266.

Edgar, Calingaert, *J. Am. Chem. Soc.*, 1929, **51**, 1546.

Wibaut *et al.*, *Rec. trav. chim.*, 1939, **58**, 329.

2 : 3 : 3-Trimethylpentane



C_8H_{18} MW, 114

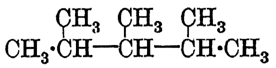
B.p. 113·6°/760 mm. (114·6°). D_4^{20} 0·7258. n_D^{20} 1·4074.

Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, **55**, 2608.

Herr, Whitmore, Schiessler, *J. Am. Chem. Soc.*, 1945, **67**, 2062.

Buck *et al.*, *J. Inst. Petroleum*, 1948, **34**, 339, (*Chem. Abstracts*, 1948, **42**, 7012).

2 : 3 : 4-Trimethylpentane



C_8H_{18} MW, 114

B.p. 112·8°/760 mm., 111·5°/732 mm. D_4^{20} 0·7197. n_D^{20} 1·4045.

Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, **55**, 2608.

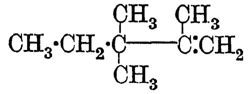
Trimethylpentanol.

See Methylethyl-*tert.*-butylcarbinol *and* Isopropyl-*tert.*-butylcarbinol.

2 : 2 : 4-Trimethylpentanone-3.

See Isopropyl-*tert.*-butyl Ketone.

2 : 3 : 3-Trimethyl-1-pentene



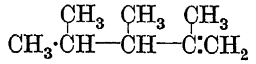
C_8H_{16} MW, 112

B.p. 108·2°/760 mm. D_4^{20} 0·7363. n_D^{20} 1·4178.

Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, **55**, 2608.

Howard *et al.*, *Journal of Research of the National Bureau of Standards*, 1947, **38**, 365, (*Chem. Abstracts*, 1947, **41**, 5290).

2 : 3 : 4-Trimethyl-1-pentene

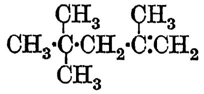


C_8H_{16} MW, 112

B.p. 107–8°/736 mm. n_D^{20} 1·4145.

Huston, Krantz, *J. Org. Chem.*, 1948, **13**, 63.

2 : 4 : 4-Trimethyl-1-pentene



C_8H_{16} MW, 112

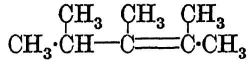
One constituent of "di-isobutylene" (or "isodibutylene"). F.p. —93·6°. B.p. 101·2°/760 mm., 100·1°/737 mm. D_4^{20} 0·7151. n_D^{20} 1·4082.

Tongberg, Pickens, Fenske, Whitmore, *J. Am. Chem. Soc.*, 1932, **54**, 3706.

Whitmore, Church, *ibid.*, 3710 (*Bibl.*).

See also Petrow, Anzus, Ardrejew, *Bull. soc. chim.*, 1933, **53**, 327 (*Bibl.*).

2 : 3 : 4-Trimethyl-2-pentene



C_8H_{16} MW, 112

B.p. 114·3°/739 mm., 52–4°/10 mm. n_D^{20} 1·4263.

Whitmore, Laughlin, *J. Am. Chem. Soc.*, 1932, **54**, 4392.

Huston, Awuapara, *J. Org. Chem.*, 1944, **9**, 401.

Howard *et al.*, *Journal of Research of the National Bureau of Standards*, 1947, **38**, 365, (*Chem. Abstracts*, 1947, **41**, 5290).

2 : 4 : 4-Trimethyl-2-pentene

$CH_3\cdot C(CH_3)_2\cdot CH{:}C(CH_3)\cdot CH_3$

C_8H_{16} MW, 112

One constituent of "di-isobutylene" (or "isodibutylene"). F.p. — 106·5°. B.p. 104·5°/760 mm., 103·4°/737 mm. D_4^{20} 0·7211. n_D^{20} 1·4158.

Tongberg, Pickens, Fenske, Whitmore, *J. Am. Chem. Soc.*, 1932, **54**, 3706.
Whitmore, Church, *ibid.*, 3710 (*Bibl.*).
Levina, Kagan, *J. Gen. Chem. U.S.S.R.*, 1941, **11**, 523.
Wibaut, van Pelt, *Rec. trav. chim.*, 1938, **57**, 1055.
See also Petrow, Anzus, Ardrejew, *Bull. soc. chim.*, 1933, **53**, 327.

3 : 4 : 4-Trimethyl-2-pentene

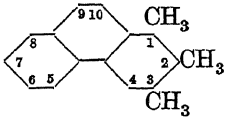
$CH_3\cdot C(CH_3)_2$——$C(CH_3){:}CH\cdot CH_3$

C_8H_{16} MW, 112

B.p. 111·9°/760 mm. D_4^{20} 0·7395. n_D^{20} 1·4232.

Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, **55**, 2608.
Henne, Matuszak, *J. Am. Chem. Soc.*, 1944, **66**, 1649.
Taylor *et al.*, *Journal of Research of the National Bureau of Standards*, 1945, **34**, 413, (*Chem. Abstracts*, 1945, **39**, 3987).

1 : 2 : 3-Trimethylphenanthrene



$C_{17}H_{16}$ MW, 220

Pale yellow needles from C_6H_6–EtOH. M.p. 64–5°.

Picrate: m.p. 187–8°.

sym.-*Trinitrobenzene add. comp.*: m.p. 201–201·5°.

Fieser, Daudt, *J. Am. Chem. Soc.*, 1941, **63**, 782.

1 : 2 : 6-Trimethylphenanthrene.

Cryst. from EtOH-pet. ether. M.p. 128·5–129°.

Picrate: red needles from EtOH. M.p. 167–8°.

Ruzicka, Rey, Smith, *Helv. Chim. Acta*, 1943, **26**, 2057.

1 : 2 : 7-Trimethylphenanthrene.

Plates from EtOH. M.p. 120–1°.

Picrate: orange needles. M.p. 148–9°.

Styphnate: orange needles. M.p. 169–70°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2250.

1 : 2 : 8-Trimethylphenanthrene.

Plates from EtOH. M.p. 144–5°. B.p. 210–20°/15 mm.

Picrate: orange-red needles from EtOH. M.p. 163°.

sym.-*Trinitrobenzene add. comp.*: yellow needles from EtOH. M.p. 193–193·5°.

Haworth, Mavin, *J. Chem. Soc.*, 1932, 2723.
Ruzicka, Hosking, *Helv. Chim. Acta*, 1931, **14**, 203.
Ruzicka, Sternbach, *Helv. Chim. Acta*, 1940, **23**, 129.

1 : 3 : 7-Trimethylphenanthrene.

Prisms from MeOH. M.p. 68–9°.

Picrate: pale orange needles from EtOH. M.p. 163–4°.

Styphnate: yellow needles. M.p. 160–1°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2250.

1 : 4 : 5-Trimethylphenanthrene.

M.p. 63·2–63·7°.

2 : 4 : 7-*Trinitrofluorenone add. comp.*: orange-red plates from C_6H_6–EtOH. M.p. 184–6°.

Newman, Hussey, *J. Am Chem. Soc.*, 1947, **69**, 3023.

1 : 4 : 7-Trimethylphenanthrene.

Prisms from EtOH. M.p. 72–3°.

Picrate: orange needles from MeOH. M.p. 141–2°.

Styphnate: yellow needles. M.p. 129–30°.

Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1789.

1 : 6 : 7-Trimethylphenanthrene.

Plates from EtOH. M.p. 123–4°.

Picrate: orange needles. M.p. 165–6°.

Styphnate: yellow needles. M.p. 111–12°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2251.

2 : 6 : 9-Trimethylphenanthrene.

Prisms from EtOH. M.p. 78·5°.

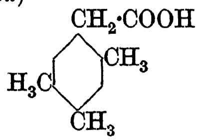
Picrate: m.p. 169–70°.

Ritter, Bogert, *J. Am. Chem. Soc.*, 1940, **62**, 1509.

Trimethylphenol.

See Hemimellitenol, Hydroxy-ψ-cumene *and* Mesitol.

2 : 4 : 5-Trimethylphenylacetic Acid (5-*ψ-Cumylacetic acid*)



$C_{11}H_{14}O_2$ MW, 178

Needles from H_2O. M.p. 128–9° (118°). Sol. usual org. solvents. Sublimes.

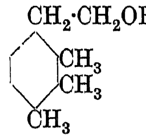
Amide: $C_{11}H_{15}ON$. MW, 177. Leaflets from H_2O. M.p. 174°. Sol. EtOH, Et_2O, $CHCl_3$. Sublimes.
Nitrile: $C_{11}H_{13}N$. MW, 159. M.p. 9–10°. B.p. 133–7°/4 mm.

Smith, MacMullen, *J. Am. Chem. Soc.*, 1936, **58**, 632.
Willgerodt, *J. prakt. Chem.*, 1909, **80**, 185.
Willgerodt, Scholz, *J. prakt. Chem.*, 1910, **81**, 388.

2 : 4 : 6-Trimethylphenylacetic Acid.
See Mesitylacetic Acid.

Trimethylphenylenediamine.
See under Phenylenediamine.

2 : 3 : 4-Trimethylphenylethyl Alcohol (2 : 3 : 4-*Trimethylbenzylcarbinol*, ω-*hydroxymethylprehnitine*)



$C_{11}H_{16}O$ MW, 164

B.p. 142–5°/9 mm.
3 : 5-*Dinitrobenzoyl*: m.p. 164–5°.
Phenylurethane: m.p. 127–8°.

Smith, Agre, *J. Am. Chem. Soc.*, 1938, **60**, 648.

Trimethylphenylglycollic Acid.
See Mesitylglycollic Acid *and* Trimethylmandelic Acid.

2 : 4 : 6-Trimethylphloroglucinol.
See 2 : 4 : 6-Trihydroxymesitylene.

Trimethyl phosphate.
See under Phosphoric Acid.

Trimethylphosphine

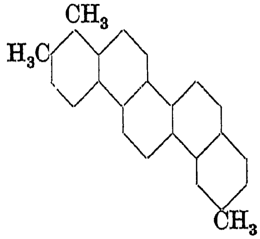
$(CH_3)_3P$

C_3H_9P **MW, 76**

B.p. 40–2°. Insol. H_2O.

Cahours, Hofmann, *Ann.*, 1857, **104**, 29.
Dreschel, *J. prakt. Chem.*, 1874, **10**, 180.

1 : 2 : 8-Trimethylpicene



$C_{25}H_{20}$ MW, 320

Leaflets from Py. M.p. 309–10°. Shows blue fluor.

Ruzicka, Hofmann, *Helv. Chim. Acta*, 1939, **22**, 126.

1 : 1 : 2-Trimethylpimelic Acid (1 : 2-*Dimethylhexane*-2 : 6-*dicarboxylic acid*)

CH3 CH3
HOOC·CH2·CH2·CH2·CH—C·COOH
CH3

$C_{10}H_{18}O_4$ MW, 202

Di-Me ester: m.p. 64–64·5°. B.p. 75–7°/0·03 mm.

Schäppi, Seidel, *Helv. Chim. Acta*, 1947, **30**, 2199.

1 : 1 : 5-Trimethylpimelic Acid (2-*Methylheptane*-2 : 6-*dicarboxylic acid*)

CH3 CH3
HOOC·CH·CH2·CH2·CH2·C·COOH
CH3

$C_{10}H_{18}O_4$ MW, 202

Cryst. from H_2O. M.p. 125°.

Colonge, Dumont, *Bull. soc. chim.*, 1947, 38.

1 : 2 : 5-Trimethylpimelic Acid (3-*Methylheptane*-2 : 6-*dicarboxylic acid*)

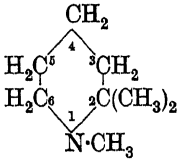
CH3 CH3 CH3
HOOC·CH·CH2·CH2·CH—CH·COOH

$C_{10}H_{18}O_4$ MW, 202

Oil. B.p. 213–15°/15 mm. Dist. with KOH ⟶ 2 : 3 : 6-trimethylcyclohexanone.

Zelinsky, Reformatsky, *Ber.*, 1895, **28**, 2944.

1 : 2 : 2-Trimethylpiperidine



$C_8H_{17}N$ MW, 127

Picrate: m.p. 270°.

Lukeš, Grossmann, *Chem. Abstracts*, 1937, **31**, 2608.

1 : 2 : 6-Trimethylpiperidine.

Cis:
Mobile liq. B.p. 50–5°/12 mm.
B,HAuCl₄: m.p. 174–5°.
Methiodide: m.p. 275°.

Mannich, *Arch. Pharm.*, 1934, **272**, 356.

2 : 2 : 4-Trimethylpiperidine.

B.p. 148°. D^{15} 0·832. Sol. EtOH, Et_2O. Mod. sol. H_2O. Turns yellow in air. $FeCl_3$ ⟶ red ppt.
B,HAuCl$_4$: prisms. M.p. 135°.
B$_2$,*H*$_2$*PtCl*$_6$: red cryst. M.p. 215–16° decomp.
Methiodide: prisms from H_2O or EtOH. M.p. 266° decomp.

Issoglio, *Chem. Zentr.*, 1908, II, 1444.

2 : 2 : 6-Trimethylpiperidine.
B.p. 138–9°.
B,HCl : m.p. 236–7°.
B,HAuCl$_4$: rhombohedra from *N*/HCl. M.p. 27–9°.
Picrate : rhombohedra from H_2O. M.p. 195–6°.

Gough, King, *J. Chem. Soc.*, 1928, 2444.

2 : 3 : 6-Trimethylpiperidine.
B.p. 36°/8 mm. D_4^{20} 0·8302. n_D^{20} 1·4434.

Nazarov, Rudenko, *Chem. Abstracts*, 1949, **43**, 2958.

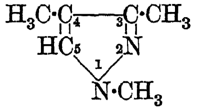
2 : 4 : 6-Trimethylpiperidine.
B.p. 165–6° (151–3°). D^4 0·8430. Misc. with EtOH, Et_2O. Spar. sol. H_2O.
B,HBr : exists in two forms. (i) Needles from H_2O. M.p. 204–9°. Sol. C_6H_6. (ii) M.p. above 270°. Spar. sol. C_6H_6.

Koenigs, Bernhart, Ibele, *Ber.*, 1907, **40**, 3199, 3206.
Skita, Brunner, *Ber.*, 1916, **49**, 1601.

2 : 2 : 6-Trimethyl-γ-piperidone.
See Vinyldiacetonamine.

Trimethylpropionic Acid.
See 1 : 2-Dimethylbutyric Acid *and tert.*-Butylacetic Acid.

1 : 3 : 4-Trimethylpyrazole



$C_6H_{10}N_2$ MW, 110

Oil. B.p. 160°. $D_4^{17·7}$ 0·9567. $n_{He}^{17·7}$ 1·48663.
Picrate : dark yellow leaflets from H_2O. M.p. 163·5–164·5°. Very spar. sol. Et_2O.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 202.

1 : 3 : 5-Trimethylpyrazole.
Needles with iodoform odour. M.p. 37°. B.p. 170°/755 mm. $D_4^{57·8}$ 0·9130. $n_{He}^{57·8}$ 1·45893.
B,HAuCl$_4$: needles + H_2O from H_2O. M.p. 91–4°.
B$_2$,*H*$_2$*PtCl*$_6$: cryst. M.p. 187–91°. Very sol. H_2O.
Picrate : m.p. 147° (144–5°).

Rojahn, Kühling, *Arch. Pharm.*, 1926, **264**, 337.
Knorr, *Ann.*, 1894, **279**, 232.

1 : 4 : 5-Trimethylpyrazole.
Oil. B.p. 176–7°. $D_4^{17·8}$ 0·9685. $n_{He}^{17·8}$ 1·48485.
Picrate : pale yellow needles from EtOH. M.p. 175–6°. Mod. sol. H_2O, EtOH, C_6H_6. Spar. sol. Et_2O.

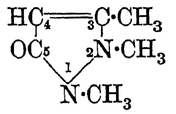
Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 202.

3 : 4 : 5-Trimethylpyrazole.
Leaflets from H_2O. M.p. 138–9°. B.p. 232–3°/753 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Volatile in steam.

B,HCl : needles. Decomp. at 265°.
B$_2$,*H*$_2$*PtCl*$_6$: yellow cryst. + $2H_2O$. Decomp. about 200°.
Picrate : yellow needles from H_2O or EtOH. M.p. 239–41°.
1-N-o-*Nitrobenzoyl* : cryst. from EtOH. M.p. 128–9°. Sol. Et_2O, Me_2CO, AcOH, C_6H_6. Spar. sol. EtOH, pet. ether.

Auwers, Cauer, Wolter, *J. prakt. Chem.*, 1930, **126**, 175.
v. Rothenburg, *J. prakt. Chem.*, 1895, **52**, 51.
Knorr, Oettinger, *Ann.*, 1894, **279**, 244.
Posner, *Ber.*, 1901, **34**, 3981.

1 : 2 : 3-Trimethylpyrazolone-5



$C_6H_{10}ON_2$ MW, 126

B.p. 306–9°/751 mm.
B$_2$,*H*$_2$*PtCl*$_6$: prisms from EtOH.Aq. Decomp. at 197–8°.
Picrate : needles from H_2O. M.p. 211–12° decomp. Spar. sol. H_2O.

Knorr, *Ber.*, 1906, **39**, 3267.

1 : 3 : 4-Trimethylpyrazolone-5.
Needles from C_6H_6. M.p. 133–4°. B.p. 154°/19 mm. Sol. EtOH, H_2O. Spar. sol. Et_2O, C_6H_6, pet. ether.
Picrate : greenish-yellow needles from EtOH. M.p. 148°.

Auwers, Bähr, *J. prakt. Chem.*, 1927, **116**, 82.

3 : 4 : 4-Trimethylpyrazolone-5.
Two compounds of this structure are described in the literature.

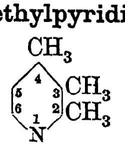
(i) Prisms from EtOH. M.p. 109·5°. Sol. H_2O, Et_2O, Me_2CO, $CHCl_3$, AcOH. Less sol. EtOH. Almost insol. pet. ether. No col. with $FeCl_3$.

Backer, *Rec. trav. chim.*, 1926, **45**, 86.

(ii) Cryst. from EtOH or H_2O. M.p. 269° (262–3°).
Acetyl : cryst. powder. M.p. 168°.

v. Rothenburg, *J. prakt. Chem.*, 1895, **52**, 43.
De, Dutt, *J. Indian Chem. Soc.*, 1930, **7**, 478.
De, Rakshit, *J. Indian. Chem. Soc.*, 1936, **13**, 509.

2 : 3 : 4-Trimethylpyridine (αβγ-*Collidine*)



$C_8H_{11}N$ MW, 121

Found in low temperature coal tar. B.p. 192–3°. D^{15} 0·9127 (0·9566). Sol. H_2O, EtOH, Et_2O. $KMnO_4 \longrightarrow$ carbocinchomeronic acid. Easily decomp.

B_2,H_2PtCl_6 : yellow prisms from H_2O. M.p. 259°.

$B,HAuCl_4$: yellow needles from H_2O. M.p. about 182–3°.

Picrate : yellow needles. M.p. 163–4°.

Guareschi, *Chem. Zentr.*, 1900, I, 1161.
Oparina, *Ber.*, 1931, **64**, 563, 574.
Prelog, Komzak, Moor, *Helv. Chim. Acta*, 1942, **25**, 1654.

2 : 3 : 5-Trimethylpyridine (*αββ'-Collidine*).
Found in low temperature coal tar. B.p. 182–3°/739 mm. Spar. sol. H_2O. Volatile in steam.

$B,HAuCl_4$: needles from H_2O. M.p. 146–7°.

B_2,H_2PtCl_6 : yellow needles from dil. HCl. M.p. 227–8°.

Picrate : yellow needles. M.p. 183° (179°).

Komatsu, Mohri, *J. Chem. Soc. Japan*, 1931, **52**, 722.
Oparina, *Ber.*, 1931, **64**, 563.

2 : 3 : 6-Trimethylpyridine (*αβα'-Collidine*).
Found in low temperature coal tar. B.p. 176–8°/759 mm., 173–4°/734 mm.

$B,HAuCl_4$: needles. M.p. 106°.

B_2,H_2PtCl_6 : cryst. + H_2O. M.p. 250–2° decomp.

Picrate : yellow needles. M.p. 146°.

Oparina, *Ber.*, 1931, **64**, 573.
Eckert, Loria, *Monatsh.*, 1917, **38**, 228, 240.
Basu, Banerjee, *J. Indian Chem. Soc.*, 1935, **12**, 665.
Hackmann, Wibaut, Gitzels, *Rec. trav. chim.*, 1943, **62**, 229.

2 : 4 : 5-Trimethylpyridine (*αγβ'-Collidine*).
B.p. 165–8°. Spar. sol. H_2O. $KMnO_4 \longrightarrow$ berberonic acid.

B_2,H_2PtCl_6,H_2O : red cryst. Decomp. at 205°.

$B,HAuCl_4$: plates or needles from dil. HCl. M.p. 129–31°.

Picrate : orange needles. M.p. 128–31°.

Ahrens, *Ber.*, 1896, **29**, 2998.

2 : 4 : 6-Trimethylpyridine (*γ-Collidine, αγα'-collidine*).
Found in low temperature coal tar. B.p. 171–2° (175–8°). D^{20} 0·917. $k = 2{\cdot}05 \times 10^{-7}$ at 25°.

B,HI : prisms. Decomp. at 250°.

B_2,H_2SO_4 : needles or prisms. M.p. 205°.

$B,HAuCl_4$: needles + H_2O. M.p. 53°, anhyd. 112–13°.

B_2,H_2PtCl_6 : yellow cryst. from H_2O. M.p. 223–4° decomp.

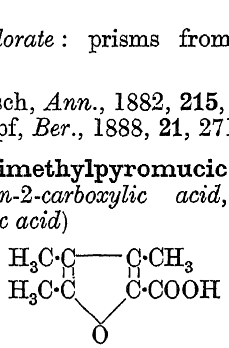
$B,HCl,2HgCl_2$: needles from H_2O. M.p. 158–60°

Picrate : yellow needles from H_2O. M.p. 155–6°.

Methoperchlorate : prisms from H_2O. M.p. 206–7°.

Hantzsch, *Ann.*, 1882, **215**, 32.
Dürkopf, *Ber.*, 1888, **21**, 2713.

3 : 4 : 5-Trimethylpyromucic Acid (*3 : 4 : 5-Trimethylfuran-2-carboxylic acid, 3 : 4 : 5-trimethyl-α-furoic acid*)

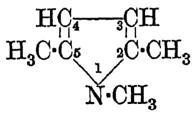


$C_8H_{10}O_3$ MW, 154

Cryst. from pet. ether. M.p. 185° decomp.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, **15**, 1117.

1 : 2 : 5-Trimethylpyrrole



$C_7H_{11}N$ MW, 109

B.p. 169°/746 mm. (162–4°), 55–6°/9 mm. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. H_2O. Volatile in steam. Hot aq. $FeCl_3 \longrightarrow$ deep cherry-red col.

Lukeš, *Chem. Abstracts*, 1932, **26**, 4328.
Lukeš, Přeučil, *Chem. Zentr.*, 1936, I, 2082.
Knorr, *Ann.*, 1886, **236**, 304.
Timoshevskaya, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 766, (*Chem. Abstracts*, 1940, **34**, 423).

2 : 3 : 4-Trimethylpyrrole.
Prisms. M.p. 39°. B.p. 71–72·5°/10 mm. Volatile in steam.

Picrate : yellow leaflets from EtOH. M.p. 142° (140°).

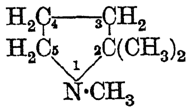
Piloty, Hirsch, *Ann.*, 1913, **395**, 66.
Fischer, Walach, *Ann.*, 1926, **450**, 114; 1926, **447**, 47.
Fischer, Neumann, Hirshbeck, *Z. physiol. Chem.*, 1943, **279**, 1.

2 : 3 : 5-Trimethylpyrrole.
B.p. 180°/768 mm., 79–80°/15 mm.

$B_2,Hg(HgCl_2)_4$: m.p. 120–5° decomp. Sol. AcOH. Spar. sol. H_2O. Insol. most org. solvents.

Korschun, *Ber.*, 1905, **38**, 1129.
Nenitzescu, Solomonica, *Ber.*, 1931, **64**, 1928.
Fischer, Müller, *Z. physiol. Chem.*, 1925, **148**, 155.
Piloty, Hirsch, *Ann.*, 1913, **395**, 68.

1 : 2 : 2-Trimethylpyrrolidine



$C_7H_{15}N$ MW, 113

Liq. B.p. 130–5°.

Lukeš, *Chem. Abstracts*, 1931, **25**, 102.

1 : 2 : 4-Trimethylpyrrolidine.

B.p. 111–13°. D^{15} 0·790.

B,HAuCl₄ : golden-yellow ppt. M.p. 98–9°.

B_2,H_2PtCl_6 : orange-yellow prisms. M.p. 179–80°. Sol. H_2O.

Jacobi, Merling, *Ann.*, 1894, **278**, 9.

1 : 2 : 5-Trimethylpyrrolidine.

B.p. 115–16°/750 mm. $D_4^{9·2}$ 0·8149. $n_\alpha^{9·2}$ 1·4335. Spar. sol. H_2O.

$B,HAuCl_4$: golden-yellow cryst. M.p. about 178°.

B_2,H_2PtCl_6 : orange-yellow leaflets from EtOH. Decomp. at 190–210°.

Picrate : cryst. from EtOH. M.p. about 163°. Spar. sol. H_2O.

Picrolonate : needles. M.p. about 193°. Sol. H_2O.

Methiodide : cryst. from EtOH. M.p. 310°.

Knorr, Rabe, *Ber.*, 1901, **34**, 3500.
Tafel, Neugebauer, *Ber.*, 1890, **23**, 1548.
Merling, *Ann.*, 1891, **264**, 334.

2 : 3 : 5-Trimethylpyrrolidine.

B.p. 126–8°. D^{15} 0·816. Misc. with H_2O.

B_2,H_2PtCl_6 : orange-red prisms. M.p. 205–6° decomp.

Jacobi, Merling, *Ann.*, 1894, **278**, 13.

Trimethylpyruvic Acid (tert.-*Butylglyoxylic acid*, 1-*keto*-2 : 2-*dimethylpropionic acid*, 1-*keto*-tert.-*butylacetic acid*)

$(CH_3)_3C·CO·COOH$

$C_6H_{10}O_3$ MW, 130

Needles from Et_2O. M.p. 90–1° (82°). B.p. 189°/747 mm., 80°/15 mm. Sol. Et_2O. Less sol. $CHCl_3$, CS_2, C_6H_6. Spar. sol. H_2O. Ox. ⟶ pivalic acid. Volatile in steam.

Me ester : $C_7H_{12}O_3$. MW, 144. B.p. 160–2°, 69–70°/20 mm. D_4^0 0·994. *Oxime* : cryst. from Et_2O–pet. ether. M.p. 66°. *Semicarbazone* : cryst. from EtOH.Aq. M.p. 125°.

Et ester : $C_8H_{14}O_3$. MW, 158. B.p. 76–7°/20 mm., 68°/15 mm. D^0 0·9716. *Oxime* : cryst. M.p. 22–3°. B.p. 131–3°/20 mm. *Semicarbazone* : cryst. from EtOH.Aq. M.p. 115°.

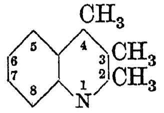
Oxime : leaflets + H_2O from EtOH. M.p. 85–6°, anhyd. 121°.

Semicarbazone : cryst. from H_2O. M.p. 195° (181°).

Phenylhydrazone : yellowish needles from EtOH.Aq. M.p. 157–8° decomp.

Richard, *Ann. chim. phys.*, 1910, **21**, 360.
Glückermann. *Monatsh.*, 1889, **10**, 771.

2 : 3 : 4-Trimethylquinoline (3 : 4-*Dimethylquinaldine*)



$C_{12}H_{13}N$ MW, 171

Cryst. M.p. 92° (65°). B.p. 285°, 156–8°/12 mm.

B,HCl : m.p. 274°. Spar. sol. EtOH.

B_2,H_2PtCl_6 : reddish-yellow. M.p. 215°.

Methiodide : yellow. M.p. 260°. Spar. sol. EtOH.

Picrate : cryst. from EtOH. M.p. 216°.

v. Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 387.
Combes, *Compt. rend.*, 1888, **106**, 143.

2 : 3 : 6-Trimethylquinoline (3 : 6-*Dimethylquinaldine*).

Cryst. from ligroin. M.p. 86–7°. B.p. 285°. Sol. Et_2O. Mod. sol. EtOH. Spar. sol. C_6H_6, ligroin. Insol. H_2O.

Picrate : yellow leaflets from EtOH. M.p. 212° decomp.

v. Miller, *Ber.*, 1890, **23**, 2268.

2 : 3 : 8-Trimethylquinoline (3 : 8-*Dimethylquinaldine*).

Found in Californian petroleum. Plates from 50% EtOH. M.p. 55–6°. B.p. 280°/747 mm., part. decomp. Sol. most org. solvents. Spar. sol. H_2O. Volatile in steam.

B,HCl : needles from H_2O or EtOH. Darkens at 230°, decomp. at 260°.

B,HNO_3 : needles from 95% EtOH. M.p. 160·5° decomp.

B,H_2SO_4 : prisms from 95% EtOH. Decomp. at about 275°.

Picrate : yellow cryst. from AcOH. Decomp. at 242–5°.

Poth *et al.*, *J. Am. Chem. Soc.*, 1930, **52**, 1245.

2 : 4 : 6-Trimethylquinoline (4 : 6-*Dimethylquinaldine*).

Needles + H_2O from H_2O + trace EtOH. M.p. 39·4°, anhyd. 65·5°. B.p. 281–2°, 146–8°/13·5 mm. Sol. EtOH, Et_2O, $CHCl_3$, Me_2CO, C_6H_6, pet. ether. Spar. sol. H_2O. Bitter taste. Sweet odour. Spar. volatile in steam.

B,HCl : needles from H_2O. M.p. 268–72°.

B,HBr : needles from EtOH.Aq. M.p. about 265–70°.

B_2,H_2SO_4 : needles from EtOH. M.p. 221–2°.

$B,HAuCl_4$: yellow needles. M.p. 140° decomp.

Tartrate : cryst. from EtOH. M.p. 172°.

Picrate : greenish-yellow needles from Me_2CO. M.p. 200–1° (189°). Spar. sol. hot EtOH.

Picrolonate : yellow needles from C_6H_6–EtOH. M.p. about 245°.

Methiodide : pale yellow needles from EtOH. M.p. 245–7° (225–6°).
Ethochloride : needles from EtOH–Et_2O. Decomp. at 247°. Very sol. H_2O, EtOH.
Ethobromide : leaflets from EtOH. Decomp. at 246°. Very sol. H_2O.

Pfitzinger, *J. prakt. Chem.*, 1888, **38**, 41.
Knoll, D.R.Ps., 363,582–3, (*Chem. Abstracts*, 1924, **18**, 991).
Bähr, *Ber.*, 1922, **55**, 1925.
Fischer, Scheibe, Müller, Merkel, *J. prakt. Chem.*, 1920, **100**, 97.
Julia, *Compt. rend.*, 1949, **228**, 1807.
Kozlov, Olifson, *J. Gen. Chem. U.S.S.R.*, 1937, **7**, 2301, (*Chem. Abstracts*, 1938, **32**, 565).

2 : 4 : 7-Trimethylquinoline (4 : 7-*Dimethylquinaldine*).
Liq. B.p. 280–1°. D^{20} 1·0337. n_D^{24} 1·59732. Forms hydrate with $1H_2O$, m.p. 48°.
B,HCl : cryst. + $2H_2O$. Sublimes at 310°.
B,HBr : cryst. + H_2O. Decomp. at 351°.
B,HI : decomp. at 320°.
B,HAuCl₄ : m.p. 152°.
B_2,H_2PtCl_6 : decomp. at 272°.
B_2,H_2SO_4 : cryst. + H_2O. M.p. 233°.
Picrate : decomp. at 232°.
Methiodide : sublimes at 322°.

Yamaguchi, *J. Pharm. Soc. Japan*, 1924, **503**, 23.

2 : 4 : 8-Trimethylquinoline (4 : 8-*Dimethylquinaldine*).
Found in petroleum. Cryst. M.p. 42°. B.p. 269–70° (280°/746 mm.). n_D^{50} 1·5855.
B,HCl : cryst. + $2H_2O$. M.p. 238°.
B,HBr : cryst. + $2H_2O$. M.p. anhyd. 251°.
B,HI : cryst. + H_2O. M.p. 224°.
$B,HAuCl_4$: m.p. 191°.
B,H_2SO_4 : cryst. + H_2O. M.p. 263°.
B,H_2PtCl_6 : m.p. 273°.
$B_2,ZnCl_2$: m.p. 272°.
Picrate : m.p. 193°.
Methiodide : decomp. at 229°.

Perrin, Bailey, *J. Am. Chem. Soc.*, 1933, **55**, 4136.
Yamaguchi, *J. Pharm. Soc. Japan*, 1924, **503**, 23.
Kozlov, Olifson, *J. Gen. Chem. U.S.S.R.*, 1932, **7**, 2301, (*Chem. Abstracts*, 1938, **32**, 565).

2 : 6 : 8-Trimethylquinoline (6 : 8-*Dimethylquinaldine*).
Leaflets from EtOH.Aq., prisms from pet. ether. M.p. 46°. B.p. 266–7°/780 mm., 260°/719 mm. Very sol. EtOH, Et_2O, pet. ether. Insol. H_2O. Volatile in steam.
B,HCl : prisms. M.p. 207°.
B,HBr : prisms. M.p. 172–3°.
B,HI : yellow prisms. M.p. 223–4°.
B_2,H_2PtCl_6 : brown cryst. M.p. 206–7°.
Picrate : yellow prisms. M.p. 187–9° (185°).
d-*Camphorsulphonate* : m.p. 231–2°.

Panajotow, *Ber.*, 1887, **20**, 32.
v. Miller, Plöchl, *Ber.*, 1896, **29**, 1472.
Jones, Evans, *J. Chem. Soc.*, 1911, **99**, 335.
Ardashev, *J. Gen. Chem. U.S.S.R.*, 1949, **19**, 550, (*Chem. Abstracts*, 1949, **43**, 7026).

3 : 4 : 7-Trimethylquinoline.
M.p. 78°.
Picrate : cryst. from Me_2CO–MeOH. M.p. 229°.
Styphnate : m.p. 234°.

Manske, Marion, Leger, *Can. J. Research*, 1942, **20*B***, 133.

4 : 5 : 8-Trimethylquinoline.
B.p. 155°/13 mm.
Picrate : m.p. 186–7°.

Kaslow, Sommer, *J. Am. Chem. Soc.*, 1946, **68**, 646.

4 : 6 : 8-Trimethylquinoline.
M.p. 55–6°.
Picrate : m.p. 208–9°.

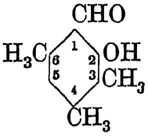
Kaslow, Sommer, *J. Am. Chem. Soc.*, 1946, **68**, 646.

5 : 6 : 8-Trimethylquinoline.
Prisms. M.p. 42–3°. B.p. 285–7°. Sol. usual solvents.
B,HNO_3 : needles. Spar. sol. H_2O.
B,H_2SO_4 : prisms from EtOH.

Berend, *Ber.*, 1885, **18**, 376.
Wikander, *Ber.*, 1900, **33**, 646.

Trimethylresorcinol.
See Dihydroxy-ψ-cumene, Dihydroxyhemimellitene *and* Mesorcinol.

3 : 4 : 6-Trimethylsalicylaldehyde (6-*Hydroxy*-2 : 4 : 5-*trimethylbenzaldehyde*)



$C_{10}H_{12}O_2$ MW, 164

Plates from EtOH. M.p. 78–9°.

Gattermann, *Ann.*, 1906, **347**, 379.

3 : 5 : 6-Trimethylsalicylaldehyde (6-*Hydroxy*-2 : 3 : 5-*trimethylbenzaldehyde*).
Yellow needles from EtOH. M.p. 105–6°. Sol. Et_2O, $CHCl_3$, AcOH. Spar. sol. cold EtOH. Insol. H_2O. Sublimes. $FeCl_3$ ⟶ blue col.

Auwers, *Ber.*, 1884, **17**, 2976.

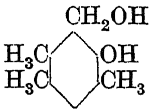
4 : 5 : 6-Trimethylsalicylaldehyde (6-*Hydroxy*-2 : 3 : 4-*trimethylbenzaldehyde*).
Pale yellow needles or prisms from MeOH. M.p. 77–8°. Sol. Me_2CO, $CHCl_3$, C_6H_6. Alc. $FeCl_3$ ⟶ intense green col.

Semicarbazone: cryst. from AcOH. Does not melt below 280°.

Auwers, Ziegler, *Ann.*, 1921, **425**, 276.

Trimethylsalicylic Acid.
See 6-Hydroxydurylic Acid *and* 6-Hydroxy-γ-isodurylic Acid.

3 : 5 : 6-Trimethylsaligenin (*6-Hydroxy-2 : 3 : 5-trimethylbenzyl alcohol*)



$C_{10}H_{14}O_2$ MW, 166

Needles from pet. ether. M.p. 91–2°. $FeCl_3$ in EtOH.Aq. ⟶ blue col. $FeCl_3$ in EtOH ⟶ bluish-green col.

α-Me ether: $C_{11}H_{16}O_2$. MW, 180. Needles from MeOH.Aq. M.p. 44–5°. Sol. usual org. solvents.

α-Acetyl: needles from AcOH.Aq. M.p. 57–8°. Sol. EtOH, C_6H_6, AcOH, pet. ether.

Diacetyl: needles from MeOH.Aq. M.p. 50–51·5°. Sol. usual org. solvents.

Manasse, *Ber.*, 1902, **35**, 3844.
Zincke, v. Hohorst, *Ann.*, 1907, **353**, 362.

Trimethylstibine (*Antimony trimethyl*)

$(CH_3)_3Sb$

C_3H_9Sb MW, 165

B.p. 82° (80·6°). Sol. EtOH, Et_2O, CS_2. Insol. H_2O. D^{15} 1·523. n_D^{15} 1·42. Oxidises in air and takes fire. Combines with O, S, and halogens to give oxide, sulphide, and halide respectively. Pptes Ag and Hg from their sols.

Hibbert, *Ber.*, 1906, **39**, 160.
Paneth, Loleit, *J. Chem. Soc.*, 1935, 371.

Trimethylsuccinic Acid (*2-Methylbutane-2 : 3-dicarboxylic acid*)

$(CH_3)_2C{\cdot}COOH$ α
$CH_3{\cdot}CH{\cdot}COOH$ β

$C_7H_{12}O_4$ MW, 160

d-.

Cryst. from H_2O. M.p. 140°. $[\alpha]_D$ + 4·83° in H_2O.

Quinine salt: m.p. 197–8°. Spar. sol. H_2O.

dl-.

Prisms from H_2O. M.p. 152° (148–9°). Sol. 10 parts H_2O at 15°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, AcOEt. Spar. sol. C_6H_6. Insol. ligroin, CS_2. $k = 3{\cdot}22 \times 10^{-4}$ at 25°. Heat of comb. C_v 5183 Cal. Spar. volatile in steam.

β-Et ester: $C_9H_{16}O_4$. MW, 188. Liq. B.p. 158°/14 mm.

Di-Et ester: $C_{11}H_{20}O_4$. MW, 216. B.p. 226°, 111°/14 mm. D_4^{20} 0·993. n_{He}^{20} 1·427.

β-Nitrile: $C_7H_{11}O_2N$. MW, 141. Needles. M.p. 126°. Very sol. EtOH, $CHCl_3$. Spar. sol. cold H_2O. Insol. ligroin.

Anhydride: cryst. M.p. 38·5° (33°). B.p. 227°/746 mm., 106–7°/15 mm. Sol. usual solvents except cold H_2O, ligroin. Heat of comb. C_v 5884·2 Cal.

Monoanilide: needles from EtOH.Aq. M.p. 134–5°. Sol. EtOH, $CHCl_3$. Insol. ligroin.

Mono-p-toluidide: plates from EtOH. M.p. 126°. Sol. EtOH, Et_2O, $CHCl_3$, AcOH. Mod. sol. hot C_6H_6. Spar. sol. hot H_2O. Insol. ligroin.

Mono-2-naphthalide: prisms from EtOH.Aq. M.p. 153°.

Bone, Perkin, *J. Chem. Soc.*, 1895, **67**, 427.
Auwers, Oswald, *Ann.*, 1895, **285**, 260, 283, 298.
Paolini, *Gazz. chim. ital.*, 1900, **30**, ii, 508.
Bone, Sprankling, *J. Chem. Soc.*, 1899, **75**, 858.
v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 186.

Trimethylsulphonium bromide

$(CH_3)_3SBr$

C_3H_9BrS MW, 157

Cryst. from H_2O. Decomp. at 172°. M.p. 201–2° in sealed tube. Reacts neutral in aq. sol.

Steinkopf, Müller, *Ber.*, 1923, **56**, 1929.

Trimethylsulphonium chloride

$(CH_3)_3SCl$

C_3H_9ClS MW, 112·5

Cryst. Decomp. at 100°. Very sol. EtOH. Very hygroscopic.

Blättler, *Monatsh.*, 1919, **40**, 420.

Trimethylsulphonium iodide

$(CH_3)_3SI$

C_3H_9IS MW, 204

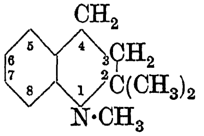
Cryst. from EtOH. Decomp. at 203–7°.

Steinkopf, Müller, *Ber.*, 1923, **56**, 1928.
Mussgnug, *Naturwiss.*, 1941, **29**, 256.

Trimethyltetrahydrobenzaldehyde.
See Cyclocitral.

1 : 1 : 6 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydronaphthalene.
See Ionene.

1 : 2 : 2 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline



$C_{12}H_{17}N$ MW, 175

B.p. 269–70°/745 mm. n_D^{19} 1·5823. Volatile in steam.

Picrate: m.p. 178°.

Freund, Richard, *Ber.*, 1909, **42**, 1112.

1 : 2 : 4 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
B.p. about 250°/759 mm.
Picrate: yellow prisms. M.p. 126–7°.
Methiodide: cryst. from EtOH. M.p. 215°.

Ciamician, Piccinini, *Ber.*, 1896, **29**, 2468.
Fischer, Meyer, *Ber.*, 1890, **23**, 2633.

1 : 2 : 6-Trimethyl-1 : 2 : 3 : 4-tetrahydroquinoline.
B.p. 145°/20 mm. D_4^{20} 0·9906. n_D^{20} 1·5611.
B,HI: m.p. 187·5–188·5°.
Picrate: cryst. from. EtOH. M.p. 141–2°.

Emerson, Davis, *J. Am. Chem. Soc.*, 1939, **61**, 2830.

1 : 2 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
Liq. B.p. 242–5°, 130°/21 mm. D_4^{20} 0·9826. n_D^{20} 1·5450.
B,HI: m.p. 154·5–155·5°.
Picrate: m.p. 177–8°.

Doebner, v. Miller, *Ber.*, 1883, **16**, 2470.
Emerson, Davis, *J. Am. Chem. Soc.*, 1939, **61**, 2830.

1 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
B,HI: prisms from MeOH–Et_2O. M.p. 164–5°.

Ewins, *J. Chem. Soc.*, 1913, **103**, 104.

2 : 2 : 4 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
Needles. M.p. 41°. B.p. 119–22°/10 mm. $D^{65·4}$ 0·9531. $n_{He}^{65·4}$ 1·53592.

Reddelien, Thurm, *Ber.*, 1932, **65**, 1520.

2 : 4 : 6 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
B.p. 265–6°/758 mm. (261·5°/762·5 mm.). D_4^{31} 0·97548. n_D^{31} 1·544354.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

2 : 4 : 7 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
B.p. 269–70°. D_4^{20} 0·9857. n_D^{20} 1·55591.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

2 : 4 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
B.p. 260–1°. D_4^{40} 0·98639. n_D^{20} 1·55598.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

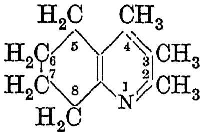
2 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
Plates. M.p. 50–51°. B.p. 260–1°/780 mm., 142–3°/14 mm.
B,HCl: prisms from H_2O. M.p. 208–9°.
B,HBr: prisms. M.p. 222–3°.
B_2,H_2PtCl_6: brown prisms. M.p. 210°.
Picrate: yellow prisms from EtOH. M.p. 179°.
N-*Acetyl*: needles from EtOH. M.p. 108–9°.
N-*Benzoyl*: prisms from EtOH. M.p. 143°.
N-*Nitroso*: yellowish needles from EtOH. M.p. 68–9°.

Jones, Evans, *J. Chem. Soc.*, 1911, **99**, 335.

5 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetrahydroquinoline.
Oil. B.p. 287–90°.
B,HCl: needles. M.p. about 238° decomp. Mod. sol. H_2O.

Wikander, *Ber.*, 1900, **33**, 648.

2 : 3 : 4 - Trimethyl - 5 : 6 : 7 : 8 - tetrahydroquinoline



$C_{12}H_{17}N$ MW, 175

B.p. 145–7°/13 mm. In moist air ⟶ dihydrate, m.p. 32°.
B,HCl: m.p. 174°. Hygroscopic.
Methiodide: m.p. 125–6°.
Picrate: m.p. 147°. Spar. sol. EtOH.

v. Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 387.

2 : 4 : 6 - Trimethyl - 5 : 6 : 7 : 8 - tetrahydroquinoline.
B.p. 256–7°. D_4^{14} 0·9845. n_D^{14} 1·53007.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

2 : 4 : 7 - Trimethyl - 5 : 6 : 7 : 8 - tetrahydroquinoline.
Cryst. M.p. 20–1°. B.p. 259–60°. $D_4^{26·5}$ 0·97559. $n_D^{26·5}$ 1·52111.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

2 : 4 : 8 - Trimethyl - 5 : 6 : 7 : 8 - tetrahydroquinoline.
B.p. 251–2°. D_4^{14} 0·99196. n_D^{14} 1·53575.

Yamaguchi, *Chem. Abstracts*, 1927, **21**, 2696.

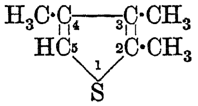
2 : 6 : 7 - Trimethyl - 5 : 6 : 7 : 8 - tetrahydroquinoline.
B.p. 239–40°/752 mm. n^{31} 1·5212.
B_2,H_2PtCl_6: m.p. 187°.
Picrate: cryst. from EtOH.Aq. M.p. 105°.

Basu, *Ann.*, 1934, **514**, 295.

1 : 1 : 4-Trimethyltetramethylene Glycol.
See 2-Methylhexandiol-2 : 5.

Trimethyltetrolic Acid.
See *tert.*-Butylpropiolic Acid.

2 : 3 : 4-Trimethylthiophene



$C_7H_{10}S$ MW, 126

Liq. B.p. 160–3°.

v. Meyer, *Die Thiophengruppe*, 60.
Zelinsky, *Ber.*, 1887, **20**, 2025.

2 : 3 : 5-Trimethylthiophene.

B.p. 163–5°/746 mm. D_4^{20} 0·9753. n_D^{20} 1·5131.

Youtz, Perkins, *J. Am. Chem. Soc.*, 1929, **51**, 3514.

Trimethylthiourea.
See under Thiourea.

ω-Trimethyltoluene.
See tert.-Butylbenzene.

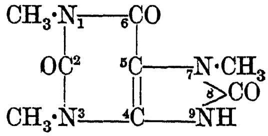
1 : 1 : 2-Trimethyltricarballylic Acid.
See Camphoronic Acid.

2 : 4 : 6-Trimethyl-1 : 3 : 5-trioxan.
See Paraldehyde.

1 : 3 : 5-Trimethyluracil.
See under Thymine.

Trimethylurea.
See under Urea.

1 : 3 : 7-Trimethyluric Acid



$C_8H_{10}O_3N_4$ MW, 210

Needles from H_2O. M.p. 345° (335°) decomp. $k = 2{\cdot}9 \times 10^{-5}$ at 25°. Spar. sol. cold H_2O, EtOH, Et_2O.

Biltz, Pardon, *J. prakt. Chem.*, 1934, **140**, 220.
Biltz, Heyn, *Ann.*, 1917, **413**, 179.

1 : 3 : 9-Trimethyluric Acid.

Plates. Decomp. at 347° (340°). Sol. NH_3. Spar. sol. EtOH, $CHCl_3$. Insol. H_2O. Sol. 30 parts boiling H_2O. $k = 1{\cdot}3 \times 10^{-6}$ at 25°. Reduces $NH_3.AgNO_3$ on boiling.

Biltz, Strufe, *Ann.*, 1921, **423**, 242.
Biltz, Pardon, *Ann.*, 1935, **515**, 241; *Ber.*, 1930, **63**, 2878.

1 : 7 : 9-Trimethyluric Acid.

Needles from EtOH or H_2O. M.p. 345° (338°). Mod. sol. hot H_2O. Spar. sol. hot EtOH, $CHCl_3$. $k = 1{\cdot}00 \times 10^{-4}$ at 25°. Sublimes.

Biltz, Krzikalla, *Ann.*, 1921, **423**, 180.

3 : 7 : 9-Trimethyluric Acid.

Needles. M.p. 373–5°. $k = 1{\cdot}7 \times 10^{-6}$ at 25°.

Biltz, Damm, *Ann.*, 1917, **413**, 186.
Biltz, Pardon, *Ber.*, 1930, **63**, 2876.

Trimyristin (*Glycerol trimyristate*)

$CH_2{\cdot}O{\cdot}CO{\cdot}[CH_2]_{12}{\cdot}CH_3$
$CH{\cdot}O{\cdot}CO{\cdot}[CH_2]_{12}{\cdot}CH_3$
$CH_2{\cdot}O{\cdot}CO{\cdot}[CH_2]_{12}{\cdot}CH_3$

$C_{45}H_{86}O_6$ MW, 722

Found in nutmegs. Exists in polymorphous forms. (i) Unstable. M.p. 32·1°. (ii) Unstable. M.p. 41·8°. (iii) Stable. Plates from Et_2O. M.p. 56·5°.

Sol. Et_2O, $CHCl_3$, C_6H_6, pet. ether. Spar. sol. EtOH, CS_2, ligroin. D_4^{60} 0·8848. n_D^{60} 1·44285.

Beal, *Organic Syntheses*, Collective Vol. I, 524.
Bömer, Engel, *Chem. Abstracts*, 1929, **23**, 4676.

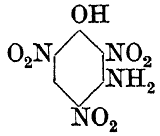
Tri-*peri*naphthylenebenzene.
See Decacyclene.

2 : 4 : 6-Trinitroacetanilide.
See under Picramide.

Trinitroaminoanisole.
See under Trinitroaminophenol.

Trinitroaminophenetole.
See under Trinitroaminophenol.

2 : 4 : 6-Trinitro-*m*-aminophenol



$C_6H_4O_7N_4$ MW, 244

Cryst. from C_6H_6. M.p. 178–9°.

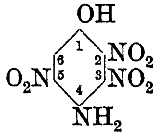
Me ether: 2 : 4 : 6-trinitro-*m*-aminoanisole, 2 : 4 : 6-trinitro-*m*-anisidine. Cryst. from MeOH. M.p. 131°.

Et ether: 2 : 4 : 6-trinitro-*m*-aminophenetole, 2 : 4 : 6-trinitro-*m*-anisidine. Cryst. from AcOH. M.p. 107°.

N-*Me*: Yellow cryst. M.p. 158°.

v. Duin, v. Lennep, *Rec. trav. chim.*, 1920, **39**, 149.
Flürscheim, D.R.P., 243,079, (*Chem. Zentr.*, 1912, I, 620).

2 : 3 : 5-Trinitro-*p*-aminophenol



$C_6H_4O_7N_4$ MW, 244

Me ether: 2 : 3 : 5-trinitro-*p*-aminoanisole, 2 : 3 : 5-trinitro-*p*-anisidine. Reddish-brown needles from H_2O. M.p. 127–8°. Sol. Me_2CO, $PhNO_2$. Mod. sol. Et_2O, C_6H_6. N-*Acetyl*: needles from AcOH.Aq. or Me_2CO.Aq. M.p. 242°. Spar. sol. boiling H_2O. Insol. Et_2O, C_6H_6. N-*Benzoyl*: m.p. 220–30°. N-p-*Toluenesulphonyl*: needles from Me_2CO. M.p. 217°.

Et ether: 2 : 3 : 5-trinitro-*p*-aminophenetole, 2 : 3 : 5-trinitro-*p*-phenetidine. Red needles

with green metallic reflex from MeOH. M.p. 126–7°. Sol. Me_2CO, hot EtOH, AcOH, $PhNO_2$. N-*Acetyl*: needles. M.p. about 245°. Sol. Me_2CO, AcOH. Spar. sol. hot EtOH, C_6H_6.

N-*Acetyl*: brownish cryst. from AcOH. M.p. 191–2°. Very sol. EtOH, AcOH.

Reverdin, Meldola, *J. prakt. Chem.*, 1913, **88**, 787.
Reverdin, Fürstenberg, *ibid.*, 323.
Reverdin, *Ber.*, 1911, **44**, 2364.

2 : 3 : 6-Trinitro-*p*-aminophenol.

Red needles from AcOH. Decomp. about 145°.

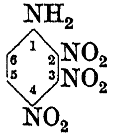
Me ether: 2 : 3 : 6-trinitro-*p*-aminoanisole, 2 : 3 : 6-trinitro-*p*-anisidine. Dark red cryst. from EtOH. M.p. 138–9°. N-*Acetyl*: needles from EtOH. M.p. 194°. N-*Benzoyl*: needles from AcOH.Aq. M.p. 205°.

N-*Acetyl*: yellow needles from AcOH. M.p. 178–9° decomp. Sol. hot EtOH. Spar. sol. hot H_2O.

N-*Propionyl*: yellowish-brown needles from AcOH. M.p. 178–9°.

Meldola, Reverdin, *J. Chem. Soc.*, 1913, **103**, 1484.
Meldola, Hay, *J. Chem. Soc.*, 1909, **95**, 1380.
Meldola, Kuntzen, *J. Chem. Soc.*, 1910, **97**, 455.

2 : 3 : 4-Trinitroaniline



$C_6H_4O_6N_4$ MW, 228

N-*Di-Me*: $C_8H_8O_6N_4$. MW, 256. Orange rhombohedra from Me_2CO or C_6H_6. M.p. 154°. D^{17} 1·551.

v. Romburgh, *Rec. trav. chim.*, 1887, **6**, 253.

2 : 4 : 5-Trinitroaniline.

Pale yellow needles from AcOH.

N-*Di-Me*: red cryst. from $Me_2CO–C_6H_6$. M.p. 196°. D^{17} 1·585.

Witt, Witte, *Ber.*, 1908, **41**, 3095.
v. Romburgh, *Rec. trav. chim.*, 1887, **6**, 253.

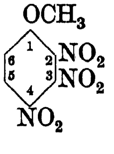
2 : 4 : 6-Trinitroaniline.

See Picramide.

Trinitroanisidine.

See under Trinitroaminophenol.

2 : 3 : 4-Trinitroanisole



$C_7H_5O_7N_3$ MW, 243

Yellow leaflets from EtOH. M.p. 155°. Sol. Me_2CO. Spar. sol. EtOH, C_6H_6. Very spar. sol. ligroin.

Vermeulen, *Chem. Zentr.*, 1912, **I**, 724.
Meldola, Eyre, *J. Chem. Soc.*, 1902, **81**, 993.

2 : 3 : 5-Trinitroanisole.

Leaflets from EtOH or HNO_3. M.p. 106·8° (104°). D^{15} 1·618.

Blanksma, *Rec. trav. chim.*, 1904, **23**, 111.

2 : 4 : 5-Trinitroanisole.

Cryst. from EtOH. M.p. 106–7°. Spar. sol. ligroin.

Vermeulen, *Rec. trav. chim.*, 1912, **31**, 102.

2 : 4 : 6-Trinitroanisole.

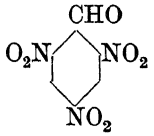
See under Picric Acid.

3 : 4 : 5-Trinitroanisole.

Cryst. M.p. 119–20°.

Vermeulen, *Rec. trav. chim.*, 1912, **31**, 103.

2 : 4 : 6-Trinitrobenzaldehyde



$C_7H_3O_7N_3$ MW, 241

Plates from C_6H_6. M.p. 119°. Conc. NH_3.Aq. in EtOH ⟶ 1 : 3 : 5-trinitrobenzene.

Oxime: cryst. from EtOH. M.p. 158°. Sol. EtOH, Me_2CO.

Semicarbazone: pale yellow leaflets from AcOH. M.p. 214° decomp.

Phenylhydrazone: reddish-brown needles. M.p. 202° (193–4°). Sol. hot Me_2CO. Spar. sol. EtOH, Et_2O, C_6H_6.

p-*Nitrophenylhydrazone*: light red needles from hot Me_2CO. M.p. 247°. Mod. sol. AcOEt, $PhNO_2$. Spar. sol. EtOH, $CHCl_3$, AcOH.

Sachs, Everding, *Ber.*, 1902, **35**, 1236; D.R.P., 121,745, (*Chem. Zentr.*, 1901, II, 69).
Secareanu, *Bull. soc. chim.*, 1932, **51**, 591; *Ber.*, 1931, **64**, 837.

2 : 4 : 6-Trinitrobenzanilide.

See under Picramide.

1 : 2 : 3-Trinitrobenzene

$C_6H_3O_6N_3$ MW, 213

Yellowish needles or prisms from MeOH. M.p. 127·5° (121°). Sol. 10 parts boiling EtOH.

Borsche, *Ber.*, 1923, **56**, 1500.
Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, II, 464.

1 : 2 : 4-Trinitrobenzene.

Pale yellow prisms from MeOH.Aq. M.p. 61–2° (57·5°). Very sol. C_6H_6. Sol. EtOH, MeOH, Et_2O, $CHCl_3$. Heat of comb. C_p 678·5 Cal.

Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, I, 634.

Borsche, *Ber.*, 1923, **56**, 1498.

1 : 3 : 5-Trinitrobenzene.

Exists in dimorphous forms. Cryst. from EtOH or HNO_3. M.ps. 122·48° and 61°. Sol. MeOH, $CHCl_3$, C_6H_6. Spar. sol. EtOH, Et_2O, CS_2. Sol. 2500 parts cold H_2O. Heat of comb. C_p 663·8 Cal.

Heinemann, B.P. 102,216, (*Chem. Abstracts*, 1917, **11**, 889).

Radcliffe, Pollitt, *J. Soc. Chem. Ind.*, 1921, **40**, 45, 90T.

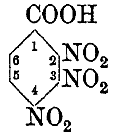
Drummond, *J. Soc. Chem. Ind.*, 1922, **41**, 338T.

Desvergnes, *Chimie et industrie*, 1931, 25, 3, 291 (*Bibl.*).

Secareanu, *Bull. soc. chim.*, 1932, **51**, 591.

Clarke, Hartman, *Organic Syntheses*, Collective Vol. I, 526.

2 : 3 : 4-Trinitrobenzoic Acid



$C_7H_3O_8N_3$ MW, 257

Prisms from H_2O or C_6H_6. M.p. 202–3°. Sol. EtOH, Me_2CO. Mod. sol. H_2O, C_6H_6. Insol. pet. ether. Explodes when heated in quantities greater than 1 gm.

Et ester: $C_9H_7O_8N_3$. MW, 285. Needles. M.p. 79–80°. Sol. Me_2CO, $CHCl_3$, C_6H_6, Et_2O. Spar. sol. pet. ether.

Giua, *Gazz. chim. ital.*, 1915, **45**, i, 348.

2 : 3 : 5-Trinitrobenzoic Acid.

Leaflets + $2H_2O$. from H_2O. M.p. 82°, anhyd. 171°.

Körner, Contardi, *Atti accad. Lincei*, 1915, **24**, I, 893.

2 : 3 : 6-Trinitrobenzoic Acid.

Fine white needles + $2H_2O$ from H_2O. M.p. 55°, anhyd. 160° decomp. Above 160° or by boiling with H_2O ⟶ 1 : 2 : 4-trinitrobenzene.

Körner, Contardi, *Atti accad. Lincei*, 1916, **25**, II, 348.

2 : 4 : 5-Trinitrobenzoic Acid.

Plates from H_2O. M.p. 194·5° decomp. Sol. EtOH, Et_2O. Mod. sol. C_6H_6. Spar. sol. pet. ether.

Me ester: $C_8H_5O_8N_3$. MW, 271. Cryst. from MeOH. M.p. 102°. Sol. usual org. solvents.

Et ester: $C_9H_7O_8N_3$. MW, 285. Leaflets from EtOH.Aq. M.p. 84°. Sol. Me_2CO, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. pet. ether.

Giua, *Gazz. chim. ital.*, 1915, **45**, i, 350.

2 : 4 : 6-Trinitrobenzoic Acid.

Rhombohedra from H_2O. M.p. 228·7° (210°). Above m.p. or with conc. NH_3.Aq. in EtOH ⟶ 1 : 3 : 5-trinitrobenzene.

Me ester: orange-yellow plates from EtOH.Aq. M.p. 160–1°.

Et ester: orange-yellow plates. M.p. 156–7°.

Propyl ester: $C_{10}H_9O_8N_3$. MW, 299. Plates. M.p. 145–6°.

Isopropyl ester: yellow plates. M.p. 154–5°.

Butyl ester: $C_{11}H_{11}O_8N_3$. MW, 313. Plates. M.p. 125–6°.

Isobutyl ester: m.p. 127–8°.

n-*Amyl ester*: $C_{12}H_{13}O_8N_3$. MW, 327. M.p. 124–5°.

Isoamyl ester: m.p. 134–5°.

Phenyl ester: $C_{13}H_7O_8N_3$. MW, 333. Cryst. from EtOH. M.p. 170·5–171·5°.

Chloride: $C_7H_2O_7N_3Cl$. MW, 275·5. Plates from C_6H_6. M.p. 163° (158°). Spar. sol. Et_2O.

Amide: $C_7H_4O_7N_4$. MW, 256. Cryst. from Me_2CO–C_6H_6–pet. ether. M.p. 264°.

Methylamide: $C_8H_6O_7N_4$. MW, 270. M.p. 285°.

Dimethylamide: $C_9H_8O_7N_4$. MW, 284. M.p. 144°.

Anhydride: $C_{14}H_4O_{15}N_6$. MW, 496. Needles. M.p. 270°.

Clarke, Hartman, *Organic Syntheses*, Collective Vol. I, 528.

Chang, Kao, *J. Chinese Chem. Soc.*, 1935, **3**, 256.

Krauz, Turek, *Chimie et industrie*, 1926, Special No., 526.

Lüttgen, D.R.P., 226,225, (*Chem. Zentr.*, 1910, II, 1174).

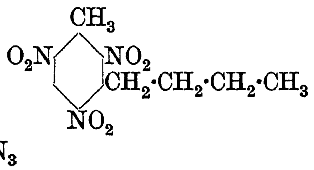
Brown, *J. Soc. Chem. Ind.*, 1947, **66**, 168.

3 : 4 : 5-Trinitrobenzoic Acid.

Greenish-yellow needles + Et_2O from Et_2O. M.p. 168° decomp.

Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, II, 467.

2 : 4 : 6-Trinitro-3-*n*-butyltoluene (*2 : 4 : 6-Trinitro-3-methyl-*n*-butylbenzene*)

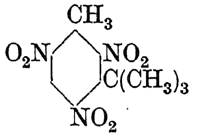


$C_{11}H_{13}O_6N_3$ MW, 283

Fine needles from EtOH. M.p. 78·5°. Odourless. Turns pink, then yellowish-brown in light.

de Capeller, *Helv. Chim. Acta*, 1928, **11**, 168.

2 : 4 : 6 - Trinitro - 3 - *tert.* - butyltoluene (2 : 4 : 6-*Trinitro*-3-*methyl*-tert.-*butylbenzene*)

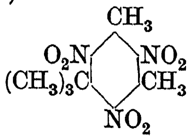


$C_{11}H_{13}O_6N_3$ MW, 283

Yellowish needles from EtOH. M.p. 96–7°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6, pet. ether. Insol. H_2O. Strong odour of musk. Non-poisonous.

Baur, *Compt. rend.*, 1890, **111**, 239.

2 : 4 : 6 - Trinitro-5-*tert.*-butyl-*m*-xylene (*Xylene musk*)

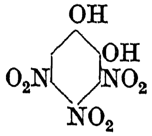


$C_{12}H_{15}O_6N_3$ MW, 297

Needles from EtOH. M.p. 110°. Possesses strong musk-like odour.

Baur, *Ber.*, 1891, 24, 2841.
Fabr. de Thann et Mulhouse, D.R.P., 77,299.

3 : 4 : 5-Trinitrocatechol

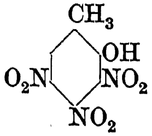


$C_6H_3O_8N_3$ MW, 245

Di-Me ether : *see* 3 : 4 : 5-Trinitroveratrol.
Di-Et ether: $C_{10}H_{11}O_8N_3$. MW, 301. Needles. M.p. 122°.

Blanksma, *Rec. trav. chim.*, 1905, **24**, 42.

3 : 4 : 5-Trinitro-*o*-cresol

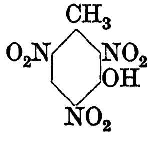


$C_7H_5O_7N_3$ MW, 243

Orange-yellow prisms from Me_2CO. M.p. 102°. Very sol. $CHCl_3$. Sol. EtOH, Et_2O, Me_2CO, AcOEt. Spar. sol. cold H_2O.
Me ether: $C_8H_7O_7N_3$. MW, 257. Needles from EtOH.Aq. M.p. 111–12°. Sol. hot EtOH.Aq., AcOH.

Sommer, *J. prakt. Chem.*, 1903, **67**, 554.
Nölting, Collin, *Ber.*, 1884, **17**, 270.

2 : 4 : 6-Trinitro-*m*-cresol

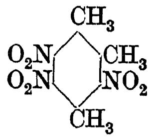


$C_7H_5O_7N_3$ MW, 243

Yellow needles from EtOH. M.p. 109–10°. Sol. 123 parts boiling and 446 parts cold H_2O. Sol. EtOH, Et_2O, Me_2CO, C_6H_6.
Me ether: $C_8H_7O_7N_3$. MW, 257. Prisms from EtOH. M.p. 94°.
Et ether: $C_9H_9O_7N_3$. MW, 271. Leaflets from EtOH. M.p. 75°. Sol. Et_2O, Me_2CO, $CHCl_3$, C_6H_6. Turns yellow in air.
Acetyl : pale yellow plates from C_6H_6. M.p. 135°.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2041.
Giua, *Gazz. chim. ital.*, 1919, **49**, ii, 164.
Beilstein, Kellner, *Ann.*, 1863, **128**, 165.
Shorygin, Topchiev, Anan'ina, *J. Gen. Chem. U.S.S.R.*, 1938, **8**, 981, (*Chem. Abstracts*, 1939, **33**, 3781).
Titov, Laptev, *J. Gen. Chem. U.S.S.R.*, 1948, **18**, 741, (*Chem. Abstracts*, 1949, **43**, 5013).

Trinitro-ψ-cumene (3 : 5 : 6-*Trinitro*-1 : 2 : 4-*trimethylbenzene*)

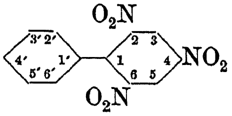


$C_9H_9O_6N_3$ MW, 255

Prisms. M.p. 185° (181–2°). Very sol. boiling C_6H_6, toluene. Spar. sol. EtOH.

Schultz, *Ber.*, 1909, **42**, 3608.
Fittig, Laubinger, *Ann.*, 1869, **151**, 261.
Migliardi, *Chem. Abstracts*, 1939, **33**, 7748.

2 : 4 : 6-Trinitrodiphenyl



$C_{12}H_7O_6N_3$ MW, 289

Pale yellowish needles from EtOH. M.p. 130°.

Gull, Turner, *J. Chem. Soc.*, 1929, 498.

2 : 4 : 2′-Trinitrodiphenyl.
Pale yellow prisms from AcOH. M.p. 150–1°.

Gull, Turner, *J. Chem. Soc.*, 1929, 498.

2 : 4 : 3′-Trinitrodiphenyl.
Cryst. from EtOH. M.p. 137–8°.

Case, *J. Am. Chem. Soc.*, 1942, **64**, 2225.

2 : 4 : 4′-Trinitrodiphenyl.
Pale yellow cubes from AcOH or HNO_3. M.p. 176°.

Gull, Turner, *J. Chem. Soc.*, 1929, 498.

3 : 4 : 3′-Trinitrodiphenyl.
Cryst. from Me_2CO–EtOH. M.p. 179–80°.

Case, *J. Am. Chem. Soc.*, 1942, **64**, 2225.

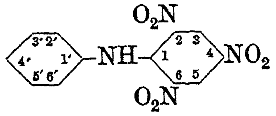
3 : 4 : 4′-Trinitrodiphenyl.
Cryst. from C_6H_6. M.p. 205–6°.

Case, *J. Am. Chem. Soc.*, 1942, **64**, 2225.

3 : 5 : 3′-Trinitrodiphenyl.
Cryst. from EtOH–Me_2CO. M.p. 173–4°.

Case, *J. Am. Chem. Soc.*, 1942, **64**, 1848.

2 : 4 : 6-Trinitrodiphenylamine (*Picrylaniline*, N-*phenylpicramide*)



$C_{12}H_8O_6N_4$ MW, 304

Yellow needles from EtOH. M.p. 179° (177°). Sol. warm AcOH, C_6H_6. Insol. ligroin.

Giua, Cherchi, *Gazz. chim. ital.*, 1919, **49**, ii, 157.
Ullmann, Nádai, *Ber.*, 1908, **41**, 1876.
Le Fèvre, *J. Chem. Soc.*, 1931, 813.

2 : 4 : 2′-Trinitrodiphenylamine.
Yellow prisms from EtOH or AcOH. M.p. 183–4°.

Juillard, *Bull. soc. chim.*, 1905, **33**, 1185.

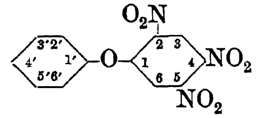
2 : 4 : 3′-Trinitrodiphenylamine.
Pale brown plates or yellow needles from AcOH. M.p. 194–5° (190°). Mod. sol. hot AcOH. Spar. sol. hot EtOH. Conc. H_2SO_4 ⟶ pale yellow sol.

v. der Kam, *Rec. trav. chim.*, 1926, **45**, 732.
Kym, Ringer, *Ber.*, 1915, **48**, 1681.

2 : 4 : 4′-Trinitrodiphenylamine.
Yellow needles from EtOH or AcOH. M.p. 189° (181°). Sol. Me_2CO. Spar. sol. AcOH, toluene.

v. der Kam, *Rec. trav. chim.*, 1926, **45**, 732.
Juillard, *Bull. soc. chim.*, 1905, **33**, 1182.
Wieland, Lecher, *Ann.*, 1912, **392**, 167.

2 : 4 : 5-Trinitrodiphenyl Ether



$C_{12}H_7O_7N_3$ MW, 305

Cryst. from EtOH. M.p. 106°. Sol. EtOH.

Westf.-Anhalt. Sprengstoff A.-G., D.R.P., 281,053, (*Chem. Zentr.*, 1915, I, 74).

2 : 4 : 6-Trinitrodiphenyl Ether (*Picric acid phenyl ether, phenyl picrate*).
Yellowish prisms from EtOH–C_6H_6. M.p. 153°. Sol. C_6H_6. Mod. sol. EtOH, $CHCl_3$, AcOH. Spar. sol. Et_2O. Insol. H_2O, ligroin.

Jackson, Earle, *Am. Chem. J.*, 1903, **29**, 213.

2 : 4 : 2′-Trinitrodiphenyl Ether.
Yellow needles from EtOH–AcOH. M.p. 137·5°.

Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, **48**, 2660.

2 : 4 : 3′-Trinitrodiphenyl Ether.
Yellow tablets from EtOH–AcOH. M.p. 136°.

Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, **48**, 2660.
Westf.-Anhalt. Sprengstoff A.-G., D.R.P., 281,053, (*Chem. Zentr.*, 1915, I, 74).

2 : 4 : 4′-Trinitrodiphenyl Ether.
Yellow tablets from EtOH–AcOH. M.p. 116° (100°).

Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, **48**, 2660.

1 : 1 : 1-Trinitroethane

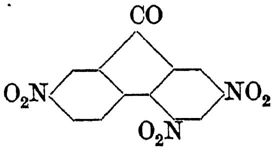
$CH_3{\cdot}C(NO_2)_3$

$C_2H_3O_6N_3$ MW, 167

Cryst. M.p. 56°. Sol. usual org. solvents. Spar. sol. H_2O. Heat of comb. C_v 1777 Cal. Very volatile.

Hantzsch, Rinckenberger, *Ber.*, 1899, **32**, 637.

2 : 4 : 7-Trinitrofluorenone

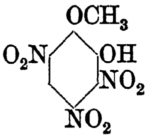


$C_{13}H_5O_7N_3$ MW, 315

Pale yellow needles from AcOH or C_6H_6. M.p. 176°. Very sol. Me_2CO, $CHCl_3$, C_6H_6.
Oxime: needles from EtOH. M.p. 260° decomp.
Semicarbazone: pale yellow. M.p. 299° decomp.
Phenylhydrazone: violet leaflets. M.p. 276° decomp.

Bell, *J. Chem. Soc.*, 1928, 1990.
Schmidt, Bauer, *Ber.*, 1905, **38**, 3760.
Woolfolk, Orchin, *Organic Syntheses*, 1948, XXVIII, 91.

3 : 4 : 6-Trinitroguaiacol



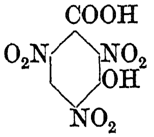
$C_7H_5O_8N_3$ MW, 259

Yellow prisms from $CHCl_3$. M.p. 129° decomp.
Me ether: *see* 3 : 4 : 6-Trinitroveratrol.

Pollecoff, Robinson, *J. Chem. Soc.*, 1918, **113**, 653.

1 : 3 : 5-Trinitrohexahydro-1 : 3 : 5-triazine.
See Cyclonite.

2 : 4 : 6-Trinitro-*m*-hydroxybenzoic Acid



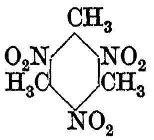
$C_7H_3O_9N_3$ MW, 273

Plates and prisms + H_2O from H_2O. M.p. about 105°. Sublimes. Blackens at 200°. Sol. H_2O, EtOH, Et_2O. Bitter taste.

Nitrile: $C_7H_2O_7N_4$. MW, 254. Yellowish needles or leaflets from dil. HNO_3. M.p. 131–2°.

Schardinger, *Ber.*, 1875, **8**, 1487.
Borsche, Gahrtz, *Ber.*, 1906, **39**, 3365.
Beilstein, Geitner, *Ann.*, 1866, **139**, 11.

Trinitromesitylene



$C_9H_9O_6N_3$ MW, 255

Needles from EtOH, prisms from Me_2CO. M.p. 235° (232°). Mod. sol. Me_2CO. Spar. sol. hot EtOH, Et_2O. Explosive.

Fittig, *Ann.*, 1867, **141**, 134.
Blanksma, *Rec. trav. chim.*, 1902, **21**, 336.

Trinitromethane (*Nitroform*)

$CH(NO_2)_3$

CHO_6N_3 MW, 151

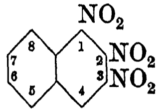
Colourless cryst. M.p. 15°. B.p. 45–7°/22 mm. $D_4^{24\cdot3}$ 1·5967. $n_{He}^{24\cdot3}$ 1·44511. Sol. H_2O with intense yellow col. Explodes on rapid heating. Forms metallic salts.

NH_4 salt: yellow needles from H_2O. Decomp. about 200°.

K salt: yellow cryst. from H_2O. Explodes at 97–9°. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O.

Chattaway, Harrison, *J. Chem. Soc.*, 1916, **109**, 171.
Hantzsch, Rinckenberger, *Ber.*, 1899, **32**, 631.

1 : 2 : 3-Trinitronaphthalene



$C_{10}H_5O_6N_3$ MW, 263

Pale orange prisms from $CHCl_3$. M.p. 190°.

Hodgson, Hathway, *J. Chem. Soc.*, 1944, 561.

1 : 2 : 4-Trinitronaphthalene.
Yellow needles from AcOH. M.p. 258°.

Contardi, Mor, *Chem. Abstracts*, 1925, **19**, 827.

1 : 2 : 5-Trinitronaphthalene.
Needles from EtOH. M.p. 112–13°.

Will, *Ber.*, 1895, **28**, 377.

1 : 3 : 5-Trinitronaphthalene.
Rhombohedra from $CHCl_3$. M.p. 122°. Sol. EtOH, AcOH. Mod. sol. $CHCl_3$.

de Aguiar, *Ber.*, 1872, **5**, 897.

1 : 3 : 6-Trinitronaphthalene.
Prisms from toluene or Me_2CO–H_2O. M.p. 186°.

Hodgson, Ward, *J. Chem. Soc.*, 1946, 533.

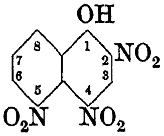
1 : 3 : 8-Trinitronaphthalene.
Cryst. from EtOH, $CHCl_3$, or AcOH. M.p. 218°. Spar. sol. Et_2O, $CHCl_3$.

Friedländer, *Ber.*, 1899, **32**, 3531.
Kalle, D.R.P., 117,368, (*Chem. Zentr.*, 1901, I, 347).
Rindl, *J. Chem. Soc.*, 1913, **103**, 1914.
Hodgson, Ward, Whitehurst, *J. Chem. Soc.*, 1945, 454.

1 : 4 : 5-Trinitronaphthalene.
Pale yellow leaflets from HNO_3. M.p. 154°. Sol. 95 parts C_6H_6, 156 parts $CHCl_3$, 260 parts Et_2O, 890 parts 90% EtOH, 4020 parts CS_2, 20,200 parts ligroin, at 18·5°.

de Aguiar, *Ber.*, 1872, **5**, 904.

2 : 4 : 5-Trinitro-1-naphthol (*Naphthopicric acid*)



$C_{10}H_5O_7N_3$ MW, 279

Yellow leaflets or prisms from AcOH. M.p. 189·5°. Sol. 364 parts cold AcOH. Spar. sol. EtOH, Et_2O, AcOEt, C_6H_6, xylene, hot H_2O.

Me ether: $C_{11}H_7O_7N_3$. MW, 293. Yellow leaflets from AcOH. M.p. 150·5–151·5° (128°). Spar. sol. EtOH, $CHCl_3$, C_6H_6.

Et ether: $C_{12}H_9O_7N_3$. MW, 307. Yellow needles from EtOH. M.p. 149–50°. Very sol. AcOH. Spar. sol. Et_2O, $CHCl_3$, C_6H_6.

Diehl, Merz, *Ber.*, 1878, **11**, 1662.
Kehrmann, Steiner, *Ber.*, 1900, **33**, 3281.
Staedel, *Ann.*, 1883, **217**, 170.
Rindl, *J. Chem. Soc.*, 1913, **103**, 1913.

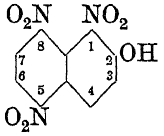
2 : 4 : 7-Trinitro-1-naphthol.
Yellow prisms from C_6H_6 or AcOH. M.p. 145°.

Kehrmann, Haberkant, *Ber.*, 1898, **21**, 2420.
Kehrmann, Steiner, *Ber.*, 1900, **33**, 3285.

2 : 4 : 8-Trinitro-1-naphthol.
Yellow prisms. M.p. 175°. Spar. sol. cold H_2O.

Graebe, *Ber.*, 1899, **32**, 2879.
Rindl, *J. Chem. Soc.*, 1913, **103**, 1916.

1 : 5 : 8-Trinitro-2-naphthol



$C_{10}H_5O_7N_3$ MW, 279

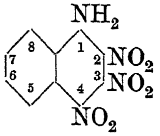
Me ether: $C_{11}H_7O_7N_3$. MW, 293. Yellow needles from AcOH. M.p. 191°.

Will, *Ber.*, 1895, **28**, 372.

1 : 6 : 8-Trinitro-2-naphthol.
Me ether: needles from AcOH. M.p. 213°. Insol. EtOH, Et_2O, $CHCl_3$, C_6H_6.
Et ether: $C_{12}H_9O_7N_3$. MW, 307. Pale yellow needles from AcOH. M.p. 186°. Spar. sol. EtOH, $CHCl_3$, C_6H_6.

Staedel, *Ann.*, 1883, **217**, 171.

2 : 3 : 4-Trinitro-1-naphthylamine



$C_{10}H_6O_6N_4$ MW, 278

Orange-yellow cryst. from EtOH–Me_2CO. M.p. 220°.

Hodgson, Hathway, *J. Chem. Soc.*, 1944, 561.

2 : 4 : 5-Trinitro-1-naphthylamine.
Yellow needles from Me_2CO. M.p. 315–17° (decomp. at 310°). Mod. sol. Me_2CO, Ac_2O. Spar. sol. most other solvents.
N-*Me*: $C_{11}H_8O_6N_4$. MW, 292. Light brown leaflets from AcOEt. M.p. 206°. Sol. Me_2CO, warm AcOH. Mod. sol. AcOEt, $CHCl_3$. Spar. sol. EtOH, Et_2O, CS_2, C_6H_6, pet. ether.
N-*Di-Me*: $C_{12}H_{10}O_6N_4$. MW, 306. Orange leaflets from AcOH. M.p. 194·5–195·5°.
N-*Et*: $C_{12}H_{10}O_6N_4$. MW, 306. Yellowish-brown needles from EtOH. M.p. 160°. Sol. Me_2CO. Mod. sol. AcOH, $CHCl_3$, C_6H_6, warm EtOH. Spar. sol. Et_2O, CS_2, pet. ether.
N-*Propyl*: $C_{13}H_{12}O_6N_4$. MW, 320. Yellow needles from AcOH. M.p. 139°.
N-*Butyl*: $C_{14}H_{14}O_6N_4$. MW, 334. Yellow needles from AcOH. M.p. 121°.
N-*Amyl*: $C_{15}H_{16}O_6N_4$. MW, 348. Yellow needles. M.p. 144–5°.
N-*Phenyl*: $C_{16}H_{10}O_6N_4$. MW, 354. Plates from AcOH. M.p. 218·5°. Mod. sol. AcOH, C_6H_6. Insol. EtOH, Et_2O, pet. ether.
N-*Acetyl*: cryst. from AcOH. Decomp. at 275°. Sol. Ac_2O. Mod. sol. AcOH.

Talen, *Rec. trav. chim.*, 1928, **47**, 355.
Groeneveld, *Rec. trav. chim.*, 1931, **50**, 692.
Rindl, *J. Chem. Soc.*, 1913, **103**, 1915.

2 : 4 : 6-Trinitro-1-naphthylamine.
Deep yellow needles from Py. M.p. 301–4°.

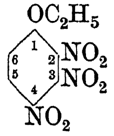
Hodgson, Turner, *J. Chem. Soc.*, 1943, 391.

1 : 6 : 8-Trinitro-2-naphthylamine.
M.p. 300° decomp. Sol. Me_2CO, AcOH, C_6H_6. Spar. sol. EtOH, Et_2O, $CHCl_3$. Insol. CS_2, pet. ether.
N-*Me*: m.p. 257° decomp.
N-*Di-Me*: pale yellow needles. M.p. 226°.
N-*Et*: orange needles. M.p. 216°.
N-*Propyl*: m.p. 186°. *Acetyl*: m.p. 179–80°.
N-*Isopropyl*: m.p. 209°.
N-*Butyl*: yellow needles. M.p. 156°.
N-*Isobutyl*: m.p. 179°.
N-*Amyl*: m.p. 181°.
N-*Isoamyl*: yellow needles. M.p. 164°.
N-*Acetyl*: pale yellow cryst. from AcOH. M.p. 239–40°. Sol. AcOH, Me_2CO. Mod. sol. EtOH, AcOEt, pet. ether.

v. der Kam, *Rec. trav. chim.*, 1926, **45**, 727.
Staedel, *Ann.*, 1883, **217**, 174.

Trinitrophenetidine.
See under Trinitroaminophenol.

2 : 3 : 4-Trinitrophenetole



$C_8H_7O_7N_3$ MW, 257

Cryst. from EtOH. M.p. 117°.

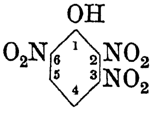
Blanksma, *Rec. trav. chim.*, 1908, **27**, 51.

2 : 3 : 5-Trinitrophenetole.
Pale yellow cryst. M.p. 80°.

Blanksma, *Rec. trav. chim.*, 1905, **24**, 41.

2 : 4 : 6-Trinitrophenetole.
See under Picric Acid.

2 : 3 : 6-Trinitrophenol



$C_6H_3O_7N_3$ MW, 229

Yellow needles from H_2O. M.p. 119°. Sol. EtOH, AcOH, C_6H_6.

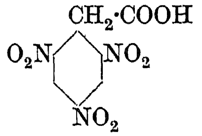
Henriques, *Ann.*, 1882, **215**, 325.
See also Reverdin, Meldola, *J. prakt. Chem.*, 1913, **88**, 796.

2 : 4 : 5-Trinitrophenol.
Needles from H_2O or EtOH.Aq. M.p. 96°. Very sol. EtOH, Et_2O, C_6H_6. Spar. sol. cold H_2O. Very bitter taste.
Me ether : *see* 2 : 4 : 5-Trinitroanisole.
Phenyl ether : *see* 2 : 4 : 5-Trinitrodiphenyl Ether.

Henriques, *Ann.*, 1882, **215**, 329.

2 : 4 : 6-Trinitrophenol.
See Picric Acid.

2 : 4 : 6-Trinitrophenylacetic Acid

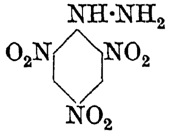


$C_8H_5O_8N_3$ MW, 271

Needles from C_6H_6. M.p. 161°. Heat with H_2O or EtOH ⟶ 2 : 4 : 6-trinitrotoluene.

Jackson, Phinney, *Am. Chem. J.*, 1899, **21**, 430.

2 : 4 : 6-Trinitrophenylhydrazine (*Picrylhydrazine*)



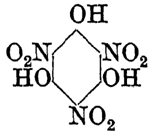
$C_6H_5O_6N_5$ MW, 243

Red plates from EtOH. M.p. 186° (175°). Sol. AcOH. Mod. sol. EtOH. Spar. sol. H_2O, Et_2O, $CHCl_3$, C_6H_6. Reduces Fehling's and $NH_3.AgNO_3$.
Acetyl : m.p. 223° (214° decomp.).

Giua, Cherchi, *Gazz. chim. ital.*, 1919, **49**, ii, 152.
Curtius, Dedichen, *J. prakt. Chem.*, 1894, **50**, 271.

2 : 4 : 6-Trinitrophenylnitramine.
See Picrylnitramine.

Trinitrophloroglucinol



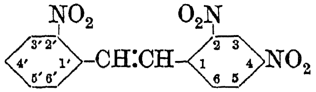
$C_6H_3O_9N_3$ MW, 261

Yellow needles + H_2O from H_2O. M.p. anhyd. 147° decomp. Sol. hot H_2O, EtOH, Et_2O. Explodes above m.p. Sublimes.
Tri-Et ether : $C_{12}H_{15}O_9N_3$. MW, 345. Plates from EtOH. M.p. 119–20°. Very sol. Me_2CO, $CHCl_3$, C_6H_6. Sol. hot EtOH, AcOH. Mod. sol. CS_2. Spar. sol. ligroin.
Triphenyl ether : $C_{24}H_{15}O_9N_3$. MW, 489. Needles from C_6H_6–EtOH. M.p. 175°. Very sol. Me_2CO, $CHCl_3$. Sol. AcOH, C_6H_6. Spar. sol. EtOH, Et_2O, CS_2, ligroin.
Tribenzyl ether : $C_{27}H_{21}O_9N_3$. MW, 531. Needles from EtOH–C_6H_6. M.p. 171°.

Freudenberg, Fikentscher, Wenner, *Ann.*, 1925, **442**, 322.
Jackson, Warren, *Am. Chem. J.*, 1893, **15**, 611.
Jackson, Smith, *Am. Chem. J.*, 1904, **32**, 173.
Blanksma, *Rec. trav. chim.*, 1908, **27**, 35.

2 : 4 : 6-Trinitroresorcinol.
See Styphnic Acid.

2 : 4 : 2′-Trinitrostilbene



$C_{14}H_9O_6N_3$ MW, 315

Greenish-yellow cryst. from AcOH. M.p. 194–5°.

Thiele, Escales, *Ber.*, 1901, **34**, 2847.

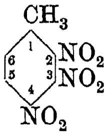
2 : 4 : 3′-Trinitrostilbene.
Yellow needles from AcOH. M.p. 183–4°. Spar. sol. boiling EtOH.

Thiele, Escales, *Ber.*, 1901, **34**, 2847.

2 : 4 : 4′-Trinitrostilbene.
Dark orange needles from AcOH. M.p. 240° (234–5°). Sol. about 70 parts boiling AcOH.

Nisbet, *J. Chem. Soc.*, 1927, 2082.
Thiele, Escales, *Ber.*, 1901, **34**, 2846.
Bayer, D.R.P., 124,681, (*Chem. Zentr.*, 1901, II, 1029).

2 : 3 : 4-Trinitrotoluene



$C_7H_5O_6N_3$ MW, 227

Prisms from Me_2CO. M.p. 112°. Sol. Et_2O, Me_2CO, C_6H_6. Mod. sol. boiling EtOH. Explodes at 290–310°.

Gornall, Robinson, *J. Chem. Soc.*, 1926, 1981.
Will, *Ber.*, 1914, **47**, 710.

2 : 3 : 5-Trinitrotoluene.
Yellowish prisms from EtOH–Et_2O. M.p. 97·5°.

Körner, Contardi, *Atti accad. Lincei*, 1915, **24**, I, 891.

2 : 3 : 6-Trinitrotoluene.
Needles from EtOH. M.p. 111°. Sol. 9 parts boiling EtOH.

Körner, Contardi, *Atti accad. Lincei*, 1916, **25**, II, 345.
Brady, Taylor, *J. Chem. Soc.*, 1920, **117**, 876.

2 : 4 : 5-Trinitrotoluene.

Yellowish plates from Me_2CO. M.p. 104°. Sol. Et_2O, Me_2CO, C_6H_6. Mod. sol. hot EtOH, AcOH. Explodes at 290–310°.

Hepp, *Ann.*, 1882, **215**, 366.
Will, *Ber.*, 1914, **47**, 710.

2 : 4 : 6-Trinitrotoluene.

Rhombohedra from EtOH. M.p. 82°. Very sol. C_6H_6. Mod. sol. Et_2O. Sol. 700 parts boiling H_2O. This compound is the explosive known as T.N.T., but the commercial product usually contains small quantities of other nitrotoluenes according to the method of manufacture.

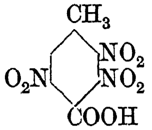
Copisarow, *Chem. News*, 1916, **113**, 37; 1915, **112**, 247.
Jackson, Phinney, *Am. Chem. J.*, 1899, **21**, 431.
Swift, Tittensor, *J. Soc. Chem. Ind.*, 1940, **59**, 92.

3 : 4 : 5-Trinitrotoluene.

Greenish-yellow prisms or plates from EtOH. M.p. 137·5°. Sol. 100 parts 95% EtOH at 15°.

Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, II, 466.

2 : 3 : 5-Trinitro-*p*-toluic Acid



$C_8H_5O_8N_3$ MW, 271

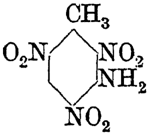
White plates. M.p. 230–1°.

Me ester: $C_9H_7O_8N_3$. MW, 285. Needles from MeOH. M.p. 114–15°.

Et ester: $C_{10}H_9O_8N_3$. MW, 299. Prisms from EtOH. M.p. 87–8°.

Giua, *Gazz. chim. ital.*, 1922, **52**, i, 183.

2 : 4 : 6-Trinitro-*m*-toluidine



$C_7H_6O_6N_4$ MW, 242

Yellow prismatic leaflets from EtOH. M.p. 138° (136°). Sol. EtOH, Et_2O.

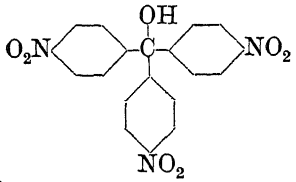
N-*Me*: $C_8H_8O_6N_4$. MW, 256. Yellow cryst. M.p. 138°.

N-*Et*: $C_9H_{10}O_6N_4$. MW, 270. Yellow cryst. M.p. 98°.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 332.
Reverdin, Dresel, Delétra, *Ber.*, 1904, **37**, 2095.

Trinitrotrimethylenetriamine.
See Cyclonite.

4 : 4′ : 4″-Trinitrotriphenylcarbinol



$C_{19}H_{13}O_7N_3$ MW, 395

Exists in two modifications.

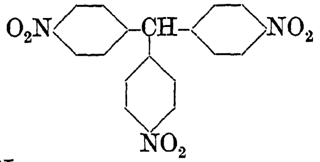
(i) Prisms from AcOH or C_6H_6. M.p. 189°.

(ii) Rhombohedra from C_6H_6 or MeOH. M.p. 167°.

Sol. AcOH, C_6H_6. Spar. sol. hot EtOH, Et_2O, CS_2. Heat of comb. C_v 2218 Cal.

Fischer, Schmidt, *Chem. Zentr.*, 1904, I, 460.
Fischer, Fischer, *Ber.*, 1904, **37**, 3355.

4 : 4′ : 4″-Trinitrotriphenylmethane

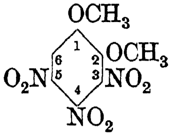


$C_{19}H_{13}O_6N_3$ MW, 379

Cryst. from C_6H_6. M.p. 212·5°. Spar. sol. AcOH, C_6H_6. Sol. alc. KOH with intense violet-blue col. Heat of comb. C_v 2272·8 Cal.

Montagne, *Rec. trav. chim.*, 1905, **24**, 125.
Hantzsch, Hein, *Ber.*, 1919, **52**, 495.

3 : 4 : 5-Trinitroveratrol



$C_8H_7O_8N_3$ MW, 273

Prisms from EtOH. M.p. 145°. Sol. hot EtOH, Et_2O. Insol. H_2O, MeOH, heptane.

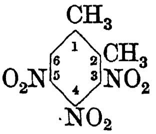
Vermeulen, *Rec. trav. chim.*, 1929, **48**, 969.
Klemenc, *Monatsh.*, 1912, **33**, 389.
Blanksma, *Rec. trav. chim.*, 1904, **23**, 114.
Frisch, Bogert, *J. Org. Chem.*, 1943, **8**, 331.

3 : 4 : 6-Trinitroveratrol.

Pale yellow needles from EtOH. M.p. 174°. Sol. Me_2CO, AcOEt. Spar. sol. EtOH, AcOH, $CHCl_3$. Decomp. slowly by cold NaOH and boiling Na_2CO_3.Aq.

Pollecoff, Robinson, *J. Chem. Soc.*, 1918, **113**, 655.

3 : 4 : 5-Trinitro-*o*-xylene



$C_8H_7O_6N_3$ MW, 241

Needles from EtOH. M.p. 115°. Sol. Me_2CO, AcOEt, $CHCl_3$, C_6H_6. Mod. sol. EtOH.

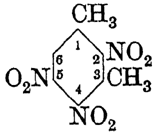
Crossley, Renouf, *J. Chem. Soc.*, 1909, **95**, 211.

3 : 4 : 6-Trinitro-*o*-xylene.

Yellowish needles from EtOH. M.p. 72°. Sol. most org. solvents except EtOH, pet. ether. Turns yellow in air.

Crossley, Renouf, *J. Chem. Soc.*, 1909, **95**, 211.

2 : 4 : 5-Trinitro-*m*-xylene



$C_8H_7O_6N_3$ MW, 241

Yellow cryst. from EtOH. M.p. 90°. D^{14} 1·553. Sol. 50 parts EtOH at 25°. Sol. HNO_3.

Blanksma, *Rec. trav. chim.*, 1909, **28**, 95; 1906, **25**, 167.

2 : 4 : 6-Trinitro-*m*-xylene.

Pale yellow prisms or leaflets from C_6H_6–EtOH. M.p. 182°. D^{19} 1·604. Sol. 2500 parts EtOH at 20°. Spar. sol. warm HNO_3.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 178.

Beilstein, Luhmann, *Ann.*, 1867, **144**, 274.

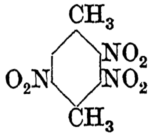
Fieser, Clapp, Daudt, *J. Am. Chem. Soc.*, 1942, **64**, 2052.

4 : 5 : 6-Trinitro-*m*-xylene.

Prisms from EtOH. M.p. 125°. D^{19} 1·494. Sol. 80 parts EtOH at 20°. Sol. warm HNO_3.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 168.

2 : 3 : 5-Trinitro-*p*-xylene



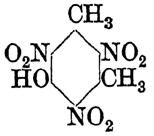
$C_8H_7O_6N_3$ MW, 241

Needles from EtOH or leaflets from EtOH–C_6H_6. M.p. 139–40° (137°). D^{19} 1·59. Turns yellow in air.

Fittig, Ahrens, Mattheides, *Ann.*, 1868, **147**, 23.

Giua, *Gazz. chim. ital.*, 1919, **49**, ii, 149 (*Note*).

2 : 4 : 6-Trinitro-*m*-5-xylenol (*Xylopicric acid*)



$C_8H_7O_7N_3$ MW, 257

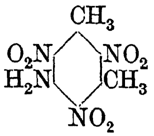
Needles from dil. HNO_3. M.p. 108° (104°). Spar. sol. H_2O with yellow col. Na, Mg, Li, Ca salts sol. H_2O. K, NH_4, Sr, Ba salts insol. H_2O.

Me ether: $C_9H_9O_7N_3$. MW, 271. Needles from EtOH. M.p. 127°. Sol. AcOH, hot EtOH. Insol. H_2O.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 329

Knecht, Hibbert, *Ber.*, 1904, **37**, 3477.

2 : 4 : 6-Trinitro-*m*-5-xylidine



$C_8H_8O_6N_4$ MW, 256

Yellow cryst. M.p. 206°.

N-*Me*: $C_9H_{10}O_6N_4$. MW, 270. Yellow cryst. M.p. 165°.

N-*Et*: $C_{10}H_{12}O_6N_4$. MW, 284. M.p. 122°.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 374; 1902, **21**, 331.

Triolein (*Olein, glycerol trioleate*)

$CH_2{\cdot}O{\cdot}CO{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}CH_3$
$CH{\cdot}O{\cdot}CO{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}CH_3$
$CH_2{\cdot}O{\cdot}CO{\cdot}[CH_2]_7{\cdot}CH{:}CH{\cdot}[CH_2]_7{\cdot}CH_3$

$C_{57}H_{104}O_6$ MW, 884

Found in olive oil, butter fat, and other vegetable oils. F.p. − 4°. B.p. 235–40°/18 mm. D_4^{60} 0·8992. n_D^{60} 1·4561. Very sol. Et_2O. Spar. sol. EtOH. Tasteless. Odourless.

Amberger, Bromig, *Biochem. Z.*, 1922, **130**, 252.

Bellucci, *Gazz. chim. ital.*, 1912, **42**, ii, 291.

Bournot, *Biochem. Z.*, 1914, **65**, 156.

Wheeler, Riemenschneider, Sando, *J. Biol. Chem.*, 1940, **132**, 687.

Trional.

See Methylsulphonal.

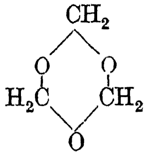
Triorsellinic Acid.

See Gyrophoric Acid.

1 : 3 : 5-Trioxan.

See Trioxymethylene.

Trioxymethylene (*Metaformaldehyde, 1:3:5-trioxan*)



$C_3H_6O_3$ MW, 90

Cryst. from Et_2O. M.p. 64° (61°). B.p. 114·5°/759 mm. Sol. EtOH, Et_2O, $CHCl_3$, CCl_4, CS_2, C_6H_6. Spar. sol. pet. ether. Sol. 5 parts H_2O at 25°. Sublimes at 46° undecomp. in presence of traces of moisture.

Diacetyl: b.p. 113–15°/2 mm. Misc. with Et_2O.

Staudinger, Lüthy, *Helv. Chim. Acta*, 1925, **8**, 51, 65.
Hammick, Boeree, *J. Chem. Soc.*, 1922, **121**, 2738.

Tripalmitin (*Palmitin, glycerol tripalmitate*)

$CH_2 \cdot O \cdot CO \cdot [CH_2]_{14} \cdot CH_3$
$CH \cdot O \cdot CO \cdot [CH_2]_{14} \cdot CH_3$
$CH_2 \cdot O \cdot CO \cdot [CH_2]_{14} \cdot CH_3$

$C_{51}H_{98}O_6$ MW, 806

Needles from Et_2O. M.p. 65·5°. D_4^{70} 0·8752. n_D^{80} 1·43807. Very sol. Et_2O. Spar. sol. EtOH.

Bellucci, *Gazz. chim. ital.*, 1912, **42**, ii, 290.
Stephenson, *Biochem. J.*, 1913, **7**, 432.
Averill, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.

Tripetroselin.
See under Petroselic Acid.

Triphenylacetic Acid (*Triphenylmethane-α-carboxylic acid*)

$(C_6H_5)_3C \cdot COOH$

$C_{20}H_{16}O_2$ MW, 288

Prisms from EtOH. M.p. 267°. Mod. sol. MeOH, EtOH, AcOH, ligroin. Spar. sol. $CHCl_3$, CS_2, C_6H_6. Very weak acid. Above m.p. part. decomp. $\longrightarrow CO_2 +$ triphenylmethane. Warm conc. $H_2SO_4 \longrightarrow CO +$ triphenylcarbinol.

Me ester: $C_{21}H_{18}O_2$. MW, 302. Needles from C_6H_6. M.p. 186°. Sol. Me_2CO, C_6H_6. Spar. sol. MeOH, Et_2O, ligroin.

Et ester: $C_{22}H_{20}O_2$. MW, 316. M.p. 120–1°.

Propyl ester: $C_{23}H_{22}O_2$. MW, 330. M.p. 98–9°.

Isopropyl ester: m.p. 84°.

Butyl ester: $C_{24}H_{24}O_2$. MW, 344. M.p. 99–99·6°.

sec.-n-*Butyl ester*: m.p. 101–2°.

Isobutyl ester: m.p. 88–9°.

n-*Amyl ester*: $C_{25}H_{26}O_2$. MW, 358. M.p. 76°.

Isoamyl ester: needles. M.p. 78·5–79·5°.

Phenyl ester: $C_{26}H_{20}O_2$. MW, 364. Needles from C_6H_6. M.p. 124·5–125°.

Benzyl ester: m.p. 99·5°.

Chloride: $C_{20}H_{15}OCl$. MW, 306·5. Prisms from C_6H_6 or ligroin. M.p. 128–9° decomp.

Amide: $C_{20}H_{17}ON$. MW, 287. Prisms from toluene. M.p. 246–7° (238°). Very spar. sol. EtOH, Et_2O.

Nitrile: α-cyanotriphenylmethane. $C_{20}H_{15}N$. MW, 269. Plates from Me_2CO.Aq. M.p. 127–8°.

Anhydride: $C_{40}H_{30}O_3$. MW, 558. Cryst. from C_6H_6. M.p. 163°.

Anilide: cryst. from AcOH. M.p. 173·5–174·5° (167–8°). N-*Benzoyl*: needles from C_6H_6. M.p. 185–6°.

Norris, Cresswell, *J. Am. Chem. Soc.*, 1934, **56**, 423.
Schlenk, Marcus, *Ber.*, 1914, **47**, 1666.
Elbs, Tölle, *J. prakt. Chem.*, 1885, **32**, 624.
Bistrzycki, Mauron, *Ber.*, 1907, **40**, 4062.
Schmidlin, Hodgson, *Ber.*, 1908, **41**, 441.
Fischer, Fischer, *Ann.*, 1878, **194**, 260.
Smith, Smith, *J. Am. Chem. Soc.*, 1948, **70**, 2400.

Triphenylacrylic Acid.
See α : β-Diphenylcinnamic Acid.

Triphenylamine

$(C_6H_5)_3N$

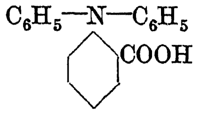
$C_{18}H_{15}N$ MW, 245

Cryst. from AcOEt. M.p. 127°. B.p. 365°. Sol. Et_2O, C_6H_6. Spar. sol. cold EtOH. Does not combine with picric acid or alkyl iodides.

Hydrochloride: m.p. 214°.

Hager, *Organic Syntheses*, Collective Vol. I, 529.
Piccard, Kharasch, *J. Am. Chem. Soc.*, 1918, **40**, 1077.

Triphenylamine-2-carboxylic Acid (*Diphenylanthranilic acid*)



$C_{19}H_{15}O_2N$ MW, 289

Yellow feathery cryst. from AcOH. M.p. 208°. Heat above m.p. $\longrightarrow$ triphenylamine. Sol. conc. H_2SO_4 with blue col.

Goldberg, Nimerovsky, *Ber.*, 1907, **40**, 2449.

Triphenylarsine (*Arsenic triphenyl*)

$(C_6H_5)_3As$

$C_{18}H_{15}As$ MW, 306

Needles from EtOH.Aq. M.p. 60·5°. B.p. above 360° in CO_2. Very sol. Et_2O, C_6H_6. Spar. sol. cold EtOH. Insol. H_2O.

B_2,H_2PtCl_6: yellow leaflets from $CHCl_3$. M.p. 285°.

Hydroxymethylate: cryst. from EtOH.Aq. M.p. 125–6°.

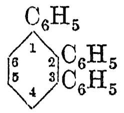
Methochloride: needles. M.p. 121°. Very sol. H_2O, EtOH.

Methiodide: yellow leaflets from EtOH. M.p. 176°.

Ethiodide: needles from EtOH–Et_2O. M.p. 158°.

Pope, Turner, *J. Chem. Soc.*, 1920, **117**, 1447.
Hilpert, Herrmann, *Ber.*, 1913, **46**, 2223.
Philips, *Ber.*, 1886, **19**, 1031.
Michaelis, *Ann.*, 1902, **321**, 160.

1 : 2 : 3-Triphenylbenzene



$C_{24}H_{18}$ MW, 306

M.p. 150–5°.

Knoevenagel, Vieth, *Ann.*, 1894, **281**, 72.

1 : 3 : 5-Triphenylbenzene.
Needles from AcOH. M.p. 172°. Distils undecomp. Sol. C_6H_6. Mod. sol. EtOH, Et_2O. CrO_3 in AcOH ⟶ benzoic acid. Causes malignant growths in mice.

Odell, Hines, *J. Am. Chem. Soc.*, 1913, **35**, 82.
Reddelien, *Ber.*, 1913, **46**, 2716.
Knoll, D.R.P., 250,236, (*Chem. Zentr.*, 1912, II, 1084).

Triphenylbenzoylmethane.
See Benzpinacolin.

Triphenyl borate.
See under Phenol.

Triphenylcarbinol (*α-Hydroxytriphenylmethane, tritanol*)

$(C_6H_5)_3C{\cdot}OH$

$C_{19}H_{16}O$ MW, 260

Plates from EtOH. M.p. 164–5°. B.p. 380°. Sol. EtOH, Et_2O, C_6H_6. Heat of comb. C_p 2342·0 Cal., C_v 2340·0 Cal. H (+ Ni) at 400°, HI in AcOH, or hot H·COOH ⟶ triphenylmethane.

Me ether: $C_{20}H_{18}O$. MW, 274. Plates from MeOH. M.p. 96–96·5°.

Et ether: $C_{21}H_{20}O$. MW, 288. Cryst. from EtOH. M.p. 84–5°.

Propyl ether: $C_{22}H_{22}O$. MW, 302. Cryst. from EtOH–Et_2O. M.p. 56°.

Isopropyl ether: m.p. 113°.

n-*Butyl ether*: b.p. 208°/5·5 mm.

Isoamyl ether: b.p. 217°/5·5 mm.

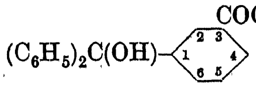
Phenyl ether: $C_{25}H_{20}O$. MW, 336. Leaflets or prisms from Et_2O–pet. ether. M.p. 103°.

Benzyl ether: plates from EtOH. M.p. 106–7°.

Acetyl: triphenylmethyl acetate. Cryst. from AcOEt–ligroin. M.p. 87–8°.

Morton, Stevens, *J. Am. Chem. Soc.*, 1931, **53**, 4030.
Stadnikow, *Ber.*, 1924, **57**, 6.
Bachmann, Hetzner, *Organic Syntheses*, 1943, XXIII, 98.
Smith, Smith, *J. Am. Chem. Soc.*, 1948, **70**, 2400.

Triphenylcarbinol-3-carboxylic Acid (*α-Hydroxytriphenylmethane-3-carboxylic acid*)



$C_{20}H_{16}O_3$ MW, 304

Cryst. from EtOH. M.p. 166–7°. Sol. conc. H_2SO_4 with yellow col.

Bistrzycki, Gyr, *Ber.*, 1904, **37**, 3698.

Triphenylcarbinol-4-carboxylic Acid (*α-Hydroxytriphenylmethane-4-carboxylic acid*).
Needles from AcOH. M.p. 203·5° (200°). Sol. EtOH, Et_2O, C_6H_6. Insol. H_2O.

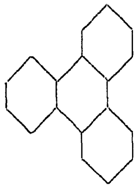
Me ester: $C_{21}H_{17}O_3$. MW, 318. Cryst. from MeOH. M.p. 119°.

Staudinger, Clar, *Ber.*, 1911, **44**, 1631.
Bistrzycki, Gyr, *Ber.*, 1904, **37**, 657.

Triphenylchloroethylene.
See Chlorotriphenylethylene.

Triphenyldihydroglyoxaline.
See Amarin.

Triphenylene (9 : 10 - *Benzphenanthrene*, 1 : 2 : 3 : 4-*dibenznaphthalene*)



$C_{18}H_{12}$ MW, 228

Occurs to a small extent in coal-tar. Needles from EtOH. M.p. 198°. Sublimes. Sol. $CHCl_3$, C_6H_6. Mod. sol. EtOH, AcOH. Sols exhibit weak blue fluor. HNO_3 at 150° ⟶ mellitic acid.

Mannich, *Ber.*, 1907, **40**, 160.
Schmidt, Schultz, *Ann.*, 1880, **203**, 135.
Bergmann, Blum-Bergmann, *J. Am. Chem. Soc.*, 1937, **59**, 1441.

1 : 1 : 1-Triphenylethane.
See α-Methyltriphenylmethane.

1 : 1 : 2-Triphenylethane

$C_6H_5{\cdot}CH_2{\cdot}CH(C_6H_5)_2$

$C_{20}H_{18}$ MW, 258

Leaflets from EtOH. M.p. 54·5°. B.p. 348–9°, 216–17°/14 mm. Forms a K salt.

Böeseken, Bastet, *Rec. trav. chim.*, 1913, **32**, 199.
Klages, Heilmann, *Ber.*, 1904, **37**, 1455.
Bert, *Compt. rend.*, 1941, **213**, 792.

Triphenylethyl Alcohol.
See Hydroxytriphenylethane.

1 : 1 : 2-Triphenylethylene (*α-Phenylstilbene*)

$C_6H_5{\cdot}CH{:}C(C_6H_5)_2$

$C_{20}H_{16}$ MW, 256

Leaflets from EtOH or AcOH. M.p. 72–3°. B.p. 220–1°/14 mm. Heat of comb. C_v 2510·3 Cal.

Stadnikow, *Ber.*, 1914, **47**, 2140.
Staudinger, Kon, *Ann.*, 1911, **384**, 89.
Adkins, Zartmann, *Organic Syntheses*, 1937, XVII, 89.

2 : 4 : 5-Triphenylglyoxaline.
See Lophine.

α-Triphenylguanidine

$$C_6H_5\cdot NH\cdot C(=N\cdot C_6H_5)\cdot NH\cdot C_6H_5$$

$C_{19}H_{17}N_3$ MW, 287

Prisms from EtOH. M.p. 144–144·5°. D^{16} 1·163. Dist. in $CO_2 \longrightarrow$ carbanilide. Conc. KOH $\longrightarrow CO_2$ + aniline. Rubber vulcanisation accelerator (T.P.G.).

B,HCl: prisms + $1H_2O$. M.p. 241–2°.

Picrate: yellow cryst. from EtOH. M.p. 180°.

Connolly, Dyson, *J. Chem. Soc.*, 1935, 680.
Hofmann, *Ber.*, 1869, **2**, 458.
Alway, Vail, *Am. Chem. J.*, 1902, **28**, 162.

β-Triphenylguanidine

$$C_6H_5\cdot NH\cdot C(=NH)\cdot N(C_6H_5)_2$$

$C_{19}H_{17}N_3$ MW, 287

Plates. M.p. 131°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6.

v. Braun, *Ber.*, 1900, **33**, 2725.
Weith, Schröder, *Ber.*, 1875, **8**, 295.

Triphenylhexahydrotriazine.
See Anhydroformaldehydeaniline.

Triphenylmethane (*Tritan*)

$$(C_6H_5)_2CH\cdot C_6H_5$$

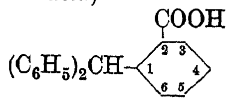
$C_{19}H_{16}$ MW, 244

Cryst. from EtOH in two forms. (i) Labile, m.p. 81°. (ii) Stable, m.p. 94°. B.p. 190–215°/10 mm. D_4^{99} 1·01405. Sol. Et_2O, $CHCl_3$, hot EtOH. Spar. sol. AcOH. Very spar. sol. ligroin. Heat of comb. C_p 2387·3 Cal., C_v 2385·1 Cal. $CrO_3 \longrightarrow$ triphenylcarbinol + benzophenone. $PCl_5 \longrightarrow$ triphenylchloromethane.

Norris, *Organic Syntheses*, Collective Vol. I, 532.
Schmidlin, Garcia-Banùs, *Ber.*, 1912, **45**, 3189.

Triphenylmethane-α-carboxylic Acid.
See Triphenylacetic Acid.

Triphenylmethane-2-carboxylic Acid (*2-Benzhydrylbenzoic acid*)



$C_{20}H_{16}O_2$ MW, 288

Needles from EtOH. M.p. 162°. Sublimes. Sol. EtOH, Et_2O, AcOH, C_6H_6. Spar. sol. ligroin. Insol. H_2O.

Me ester: $C_{21}H_{18}O_2$. MW, 302. Prisms from MeOH. M.p. 98°.

Nitrile: 2-cyanotriphenylmethane. $C_{20}H_{15}N$. MW, 269. Needles from EtOH. M.p. 89°. B.p. 270–85°/20–30 mm. Sol. EtOH, Et_2O, $CHCl_3$, AcOH, C_6H_6. Insol. ligroin.

Drory, *Ber.*, 1891, **24**, 2573.
Gresly, *Ann.*, 1886, **234**, 242.

Triphenylmethane-4-carboxylic Acid (*4-Benzhydrylbenzoic acid*).

Needles from AcOH.Aq. M.p. 165°. Sol. EtOH, Et_2O, C_6H_6. Prac. insol. H_2O.

Chloride: $C_{20}H_{15}OCl$. MW, 306·5. Cryst. from pet. ether. M.p. 89–90°.

Nitrile: 4-cyanotriphenylmethane. Prisms from MeOH. M.p. 100°. Sol. EtOH, Et_2O, AcOH, C_6H_6.

Anilide: cryst. from AcOH. M.p. 196°.

Staudinger, Clar, *Ber.*, 1911, **44**, 1628.
Moses, *Ber.*, 1900, **33**, 2630.

Triphenylmethyl.
See Hexaphenylethane.

Triphenylmethylamine.
See α-Aminotriphenylmethane.

ω-Triphenylmethyl-benzyl Alcohol.
See 1 : 2 : 2 : 2-Tetraphenylethyl Alcohol.

Triphenylmethyl bromide (*α-Bromotriphenylmethane, trityl bromide, triphenylbromomethane*)

$$(C_6H_5)_3CBr$$

$C_{19}H_{15}Br$ MW, 323

Pale yellow cryst. from CS_2. M.p. 152°. B.p. 230°/15 mm. Boiling AcOH $\longrightarrow$ triphenylcarbinol. NH_3 in C_6H_6 sol. $\longrightarrow$ triphenylmethylamine.

Allen, Kölliker, *Ann.*, 1885, **227**, 110.
Wieland, *Ber.*, 1909, **42**, 3024 (*Footnote*).

Triphenylmethyl chloride (*α-Chlorotriphenylmethane, triphenylchloromethane, trityl chloride*)

$$(C_6H_5)_3CCl$$

$C_{19}H_{15}Cl$ MW, 278·5

Cryst. from C_6H_6 or pet. ether. M.p. 112–13°. Sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Heat of comb. C_v 2346·5 Cal., C_p 2348·5 Cal. Above 250° $\longrightarrow$ triphenylmethane + a little 9-phenylfluorene. Employed for characterisation of alcohols as triphenylmethyl ethers.

Gomberg, *Ber.*, 1900, **33**, 3147.
Norris, Sanders, *Am. Chem. J.*, 1901, **25**, 60.
Bachmann, *Organic Syntheses*, 1943, XXIII, 100.

Triphenylmethyl iodide (*α-Iodotriphenylmethane, trityl iodide*)

$$(C_6H_5)_3CI$$

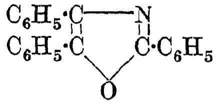
$C_{19}H_{15}I$ MW, 370

Yellowish cryst. which rapidly turn dark brown. M.p. 132°.

$C_{19}H_{15}I,5I$: prisms. M.p. 90°.

Gomberg, *Ber.*, 1900, **33**, 3158; 1902, **35**, 1835.

2 : 4 : 5-Triphenyloxazole (*Benzilam, azobenzil*)



$C_{21}H_{15}ON$ MW, 297

Prisms. M.p. 115·5–116·5°. Sol. C_6H_6 with feeble blue fluor. $CrO_3 \longrightarrow$ benzoic acid.

Schönberg, *Ber.*, 1921, **54**, 242.
McKenzie, Barrow, *J. Chem. Soc.*, 1913, **103**, 1334.
Davidson, Weiss, Jelling, *J. Org. Chem.*, 1937, **2**, 328.

Triphenyl phosphate

$(C_6H_5O)_3PO$

$C_{18}H_{15}O_4P$ MW, 326

Prisms from EtOH. M.p. 49°. B.p. 245°/11 mm. Sol. Et_2O, $CHCl_3$, C_6H_6. Mod. sol. EtOH. Insol. H_2O.

Heim, *Ber.*, 1883, **16**, 1765.
A.G.F.A., D.R.P., 246,871, (*Chem. Zentr.*, 1912, I, 1875).

Triphenylphosphine

$(C_6H_5)_3P$

$C_{18}H_{15}P$ MW, 262

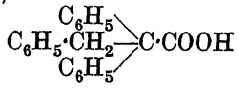
Plates or prisms from Et_2O. M.p. 80°. Dist. undecomp. above 360° in indifferent atmosphere. n_D^{89} 1·52475. Very sol. Et_2O. Sol. $CHCl_3$, AcOH, C_6H_6. Mod. sol. EtOH. Insol. H_2O. Heat of comb. C_p 2480·7 Cal., C_v 2483·95 Cal. Triboluminescent.

B,HI: needles. M.p. 215° decomp.
Methiodide: leaflets. M.p. 182–3°.
Ethiodide: leaflets from EtOH.Aq. M.p. 164–5°.
B,C₆H₅Br: tetraphenylphosphonium bromide. M.p. 286–8°.
B,C₆H₅Cl: tetraphenylphosphonium chloride. M.p. 265°.
B,C₆H₅I: tetraphenylphosphonium iodide. M.p. 333°.

Dodonow, Medox, *Ber.*, 1928, **61**, 907.
Michaelis, v. Soden, *Ann.*, 1885, **229**, 298.

Triphenyl phosphite.
See under Phenol.

1 : 1 : 2-Triphenylpropionic Acid (*Diphenylbenzylacetic acid*, 1 : 1-*diphenylhydrocinnamic acid*)



$C_{21}H_{18}O_2$ MW, 302

Needles from EtOH.Aq. M.p. 162° (132°). Sol. Et_2O, hot EtOH.Aq. Very spar. sol. H_2O.
Me ester: $C_{22}H_{20}O_2$. MW, 316. M.p. 127°.
Benzyl ester: prisms from EtOH. M.p. 85°. B.p. 270°/2 mm.
Chloride: $C_{21}H_{17}OCl$. MW, 320·5. M.p. 90–1°.
Amide: $C_{21}H_{19}ON$. MW, 301. Needles from EtOH. M.p. 111°.
Nitrile: $C_{21}H_{17}N$. MW, 283. Plates or needles. M.p. 126°.

Ramart, *Bull. soc. chim.*, 1924, **35**, 196.
Neure, *Ann.*, 1889, **250**, 143, 147.

1 : 2 : 2-Triphenylpropionic Acid (*Phenylbenzhydrylacetic acid*)

C_6H_5
$(C_6H_5)_2CH{\cdot}CH{\cdot}COOH$

$C_{21}H_{18}O_2$ MW, 302

Needles from EtOH or pet. ether. M.p. 222–3° (211°). Sol. EtOH, Et_2O. Spar. sol. ligroin. Insol. H_2O.
Me ester: plates from EtOH. M.p. 162°.
Et ester: $C_{23}H_{22}O_2$. MW, 330. Leaflets from EtOH. M.p. 122–3°.
Propyl ester: $C_{24}H_{24}O_2$. MW, 344. M.p. 109°.
Isopropyl ester: m.p. 138°.
Phenyl ester: $C_{27}H_{22}O_2$. MW, 378. M.p. 124°.
Benzyl ester: m.p. 117°.
Chloride: cryst. from pet. ether. M.p. 94°.
Amide: needles from EtOH.Aq. M.p. 213°.
Nitrile: plates from MeOH. M.p. 102°.

Kohler, Heritage, *Am. Chem. J.*, 1905, **33**, 156.
Banús, Salas, *Chem. Zentr.*, 1935, II, 3770.
Banús, Boqué, *ibid*, 3770.

2 : 2 : 2-Triphenylpropionic Acid (2 : 2-*Diphenylhydrocinnamic acid*)

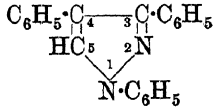
$(C_6H_5)_3C{\cdot}CH_2{\cdot}COOH$

$C_{21}H_{18}O_2$ MW, 302

Prisms from EtOH. M.p. 177°. Very sol. Et_2O. Sol. EtOH.
Et ester: prisms. M.p. 125° (81°).
Chloride: m.p. 128–9° (132°).
Amide: m.p. 198°.
Nitrile: cryst. from EtOH. M.p. 140°.

Fosse, *Bull. soc. chim.*, 1931, **49**, 159.
Henderson, *J. Chem. Soc.*, 1887, **51**, 226.

1 : 3 : 4-Triphenylpyrazole (1 : 3 : 4-*Triphenyl*-1 : 2-*diazole*)



$C_{21}H_{16}N_2$ MW, 296

Needles from EtOH. M.p. 207° (185°). Very sol. C_6H_6. Spar. sol. EtOH, ligroin, cold AcOH. Insol. dil. acids. Sol. conc. H_2SO_4 with yellow col.

Rupe, Gisiger, *Helv. Chim. Acta*, 1925, **8**, 351.

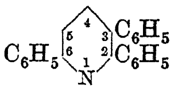
1 : 3 : 5-Triphenylpyrazole.
Needles or plates from Et_2O. M.p. 140-140·5° (137–8°). Sol. $CHCl_3$, AcOH, C_6H_6. Spar. sol. EtOH, Et_2O. Insol. H_2O.
Methiodide: needles from H_2O. M.p. 176° ⟶ components.

Knorr, Laubmann, *Ber.*, 1888, **21**, 1206.
Wislicenus, *Ann.*, 1899, **308**, 253.

1 : 4 : 5-Triphenylpyrazole.
Needles from EtOH or AcOH. M.p. 212° (206°). B.p. about 400° undecomp. Sol. $CHCl_3$, hot AcOH, hot C_6H_6. Spar. sol. Et_2O, hot EtOH.

Japp, Tingle, *J. Chem. Soc.*, 1897, **71**, 1143.
Wislicenus, Ruthing, *Ann.*, 1911, **379**, 257.

2 : 3 : 6-Triphenylpyridine



$C_{23}H_{17}N$ MW, 307

Needles from EtOH. M.p. 115°.
Picrate: yellow prisms from EtOH. M.p. 163°.

Allen, Barker, *J. Am. Chem. Soc.*, 1932, **54**, 742.

2 : 4 : 5-Triphenylpyridine.
Slightly iridescent rods from EtOH. M.p. 112°. Sol. EtOH, Et_2O, C_6H_6.

Allen, Frame, *J. Am. Chem. Soc.*, 1940, **62**, 1301.

2 : 4 : 6-Triphenylpyridine (*Acetophenine*).
Colourless prisms from EtOH. M.p. 137·5° (135°). Very spar. sol. cold EtOH. Sol. conc. H_2SO_4 with blue fluor.
Picrate: yellow needles from EtOH. M.p. 192·5°.

Reddelien, *Ber.*, 1920, **53**, 334.

Triphenylstibine (*Antimony triphenyl*)

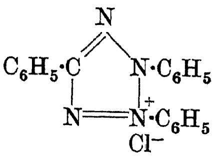
$(C_6H_5)_3Sb$

$C_{18}H_{15}Sb$ MW, 351

Prisms from pet. ether. M.p. 53° (50°). B.p. above 220°/1 mm. D_4^{50} 1·4075, D_4^{100} 1·3597. Very sol. Et_2O, $CHCl_3$, AcOH, C_6H_6, pet. ether. Spar. sol. EtOH.

Hiers, *Organic Syntheses*, Collective Vol. I, 535.

2 : 3 : 5-Triphenyltetrazolium chloride



$C_{19}H_{15}N_4Cl$ MW, 334·5

Long needles from $CHCl_3$. M.p. 243° decomp. Sol. H_2O, EtOH, Me_2CO.

Pechmann, Runge, *Ber.*, 1894, **27**, 2920.
Kuhn, Jerchel, *Ber.*, 1941, **74**, 941.

Triphenylthiourea.
See under Thiourea.

Triphenylurea.
See under Urea.

Triphosphopyridine Nucleotide.
See Coenzyme II.

Tripropin.
See under Propionic Acid.

Tripropylamine

$(CH_3{\cdot}CH_2{\cdot}CH_2)_3N$

$C_9H_{21}N$ MW, 143

Liq. B.p. 156·5°. D_4^{20} 0·753. $n_D^{19\cdot4}$ 1·41756. $k = 4{\cdot}43\ (5{\cdot}5) \times 10^{-4}$ at 25°.
B,HCl: hygroscopic needles. M.p. 90°.
B,HBr: needles. M.p. 180°.
B,HAuBr₄: m.p. 149°.
B,HBr,HgBr₂: needles. M.p. 104°.
Methiodide: methyltripropylammonium iodide. Leaflets from EtOH–Et_2O. M.p. 207–8°.
Ethiodide: ethyltripropylammonium iodide. Prisms from EtOH. M.p. 238° decomp.
B,I₂: cryst. from EtOH. M.p. 65–6°.

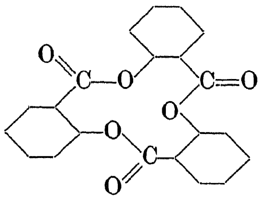
Skita, Keil, *Monatsh.*, 1929, **53** and **54**, 758.

Tripropyl phosphate.
See under Phosphoric Acid.

Triptane.
See 2 : 2-Dimethylbutane.

Tripterin.
See Celastrol.

Trisalicylide



$C_{21}H_{12}O_6$ MW, 360

Needles from C_6H_6. M.p. 200°. Formerly referred to as β-disalicylide.

Anschütz, *Ber.*, 1919, **52**, 1884; *J. prakt. Chem.*, 1922, **105**, 158.
Anschütz, Riepenkröger, *Ann.*, 1924, **439**, 1.
Anschütz, Neher, *Ber.*, 1944, **77**, 634.
Baker, Ollis, Zealley, *J. Chem. Soc.*, 1951, 201.

Trisalicylin.
See under Salicylic Acid.

Tristearin (*Glycerol tristearate, stearin*)

$$\begin{array}{l} CH_2O{\cdot}CO{\cdot}[CH_2]_{16}{\cdot}CH_3 \\ CHO{\cdot}CO{\cdot}[CH_2]_{16}{\cdot}CH_3 \\ CH_2O{\cdot}CO{\cdot}[CH_2]_{16}{\cdot}CH_3 \end{array}$$

$C_{57}H_{110}O_6$ MW, 890

Present in many natural fats. Cryst. from Et_2O. M.p. 72° (70·8°). D_4^{80} 0·8559. n_D^{80} 1·4385. Sol. $CHCl_3$, C_6H_6, hot EtOH. Mod. sol. boiling Et_2O, boiling pet. ether. Insol. cold EtOH.

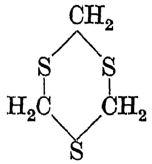
Bellucci, *Gazz. chim. ital.*, 1912, **42**, ii, 291.

Tritan.
See Triphenylmethane.

Tritanol.
See Triphenylcarbinol.

22-Tritetracontanone.
See Behenone.

Trithiane (*Trithioformaldehyde*)



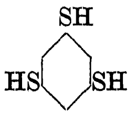
$C_3H_6S_3$ MW, 138

Needles from AcOH. M.p. 218°.

Baumann, *Ber.*, 1890, **23**, 67.
Binz, Pence, *J. Am. Chem. Soc.*, 1939, **61**, 3134.
Lee, Dougherty, *J. Org. Chem.*, 1939, **4**, 48.

Trithioformaldehyde.
See Trithiane.

Trithiophloroglucinol (1 : 3 : 5-*Trimercaptobenzene*)



$C_6H_6S_3$ MW, 174

Needles. M.p. 57–60°. Sol. Et_2O, CS_2.
Tri-Me ether: $C_9H_{12}S_3$. MW, 216. Needles from EtOH. M.p. 66–8°.
Triacetyl: needles from EtOH. M.p. 73–4°.

Pollak, Carniol, *Ber.*, 1909, **42**, 3252.

α-Tritisterol

$C_{30}H_{50}O$ MW, 426

Occurs in unsaponifiable portion of wheat germ oil. Needles from Me_2CO–MeOH. M.p. 114–15°. $[\alpha]_D^{20}$ + 54·3° in EtOH.
Acetyl: leaflets from EtOH. M.p. 107–8°. $[\alpha]_D$ + 70·4° in $CHCl_3$.
3 : 5-*Dinitrobenzoyl*: cryst. from AcOEt. M.p. 182°.
β-*Naphthoyl*: needles. M.p. 158–9°.

Karrer, Salomon, *Helv. Chim. Acta*, 1937, **20**, 424.
Karrer, Salomon, Fritzsche, *ibid.*, 1422.
Todd, Bergel, Waldmann, Work, *Biochem. J.*, 1937, **31**, 2247.
Drummond, Hoover, *ibid.*, 1852.

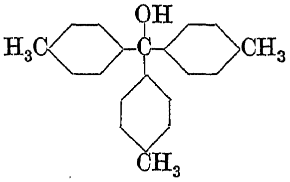
β-Tritisterol

$C_{30}H_{50}O$ MW, 426

Occurs in unsaponifiable portion of wheat germ oil. Needles from MeOH. M.p. 97°. $[\alpha]_D^{20}$ + 49·2° in EtOH.
Acetyl: needles from MeOH–EtOH. $[\alpha]_D^{20}$ + 55·5° in $CHCl_3$.
Acetyl-dibromide: cryst. from Me_2CO. M.p. 160–2°.

Karrer, Salomon, *Helv. Chim. Acta*, 1937, **20**, 424.
Karrer, Salomon, Fritzsche, *ibid.*, 1422.
Todd, Bergel, Waldmann, Work, *Biochem. J.*, 1937, **31**, 2247.
Drummond, Hoover, *ibid.*, 1852.

Tri-*p*-tolylcarbinol (α-*Hydroxy*-4 : 4′ : 4″-*trimethyltriphenylmethane*)

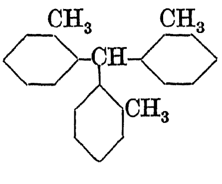


$C_{22}H_{22}O$ MW, 302

Prisms from Et_2O–ligroin. M.p. 96°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. pet. ether. $H_2SO_4 \longrightarrow$ greenish-red col.
Et ether: $C_{24}H_{26}O$. MW, 330. Needles from pet. ether. M.p. 111°.

Gomberg, *Ber.*, 1904, **37**, 1629.
Mothwurf, *ibid.*, 3153.
Tousley, Gomberg, *J. Am. Chem. Soc.*, 1904, **26**, 1516.

Tri-*o*-tolylmethane



$C_{22}H_{22}$ MW, 286

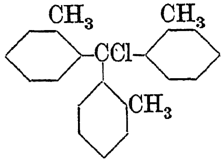
Needles from EtOH. M.p. 130·5–131·5°.

Bartlett, Jones, *J. Am. Chem. Soc.*, 1942, **64**, 1837.

Tri-*p*-tolylmethane.
Prisms. M.p. 63°. B.p. 225–30°/15 mm. Spar. sol. EtOH, AcOH.

Tousley, Gomberg, *J. Am. Chem. Soc.*, 1904, **26**, 1520.
Mothwurf, *Ber.*, 1904, **37**, 3155.

Tri-o-tolylmethyl chloride (*α-Chlorotri-*o-*tolylmethane*)



$C_{22}H_{21}Cl$ MW, 320·5

M.p. 107°.

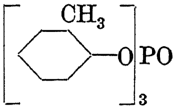
Theilacher, Ewald, *Naturwiss.*, 1943, **31**, 302.

Tri-p-tolylmethyl chloride (*α-Chlorotri-*p-*tolylmethane*).

Cryst. from AcOEt. M.p. 184–5° (173°). Sol. C_6H_6, CS_2, pet. ether. H_2SO_4 ⟶ tri-*p*-tolylcarbinol.

Mothwurf, *Ber.*, 1904, **37**, 3156.
Gomberg, *ibid.*, 1627.

Tri-o-tolyl phosphate (*Tri-*o-*cresyl phosphate*)



$C_{21}H_{21}O_4P$ MW, 368

Liq. D_4^{25} 1·183.

Breusch, Keskin, *Chem. Abstracts*, 1944, **38**, 1483.

Tri-m-tolyl phosphate (*Tri-*m-*cresyl phosphate*).

M.p. 25–6°. B.p. 258–63°/4 mm. D_4^{25} 1·150.

Breusch, Keskin, *Chem. Abstracts*, 1944, **38**, 1483.

Tri-p-tolyl phosphate (*Tri-*p-*cresyl phosphate*).

Needles from EtOH. M.p. 77·5–78°. D_4^{25} 1·247. Sol. Et_2O, EtOH, $CHCl_3$, C_6H_6, AcOH.

Breusch, Keskin, *Chem. Abstracts*, 1944, **38**, 1483.

Tritopine.
See Laudanidine.

Triuret (*Dicarbamylurea*)

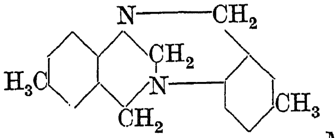
$H_2N{\cdot}CO{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}CO{\cdot}NH_2$

$C_3H_6O_3N_4$ MW, 146

Cryst. from dil. NH_3. M.p. 227° decomp. (231°, 232–4°). 5% alk. $CuSO_4$ sol. ⟶ deep blue col.

Haworth, Mann, *J. Chem. Soc.*, 1943, 603.
Werner, Gray, *Chem. Abstracts*, 1947, **41**, 710.

Tröger's Base (1 : *2'-Methylene-*3-p-*tolyl-*6-*methyl-*1 : 2 : 3 : *4-tetrahydroquinazoline*)



$C_{17}H_{18}N_2$ MW, 250

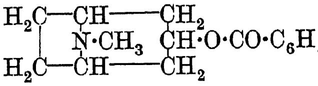
Needles from EtOH.Aq. M.p. 135–6°. Resolved into optical antipodes by chromatography.

Hydrochloride: m.p. 213°.

Picrate: cryst. from EtOH. M.p. 188–9°.

Wagner, *J. Am. Chem. Soc.*, 1935, **57**, 1296.
Spielman, *ibid.*, 583.
Prelog, Wieland, *Helv. Chim. Acta*, 1944, **27**, 1127.

Tropacocaine (*ψ-Tropyl ester of benzoic acid, benzoyl-ψ-tropeïn*)



$C_{15}H_{19}O_2N$ MW, 245

Constituent of Java coca leaves. Plates. M.p. 49°. Sol. EtOH, Et_2O, $CHCl_3$, ligroin, C_6H_6. Hot conc. HCl ⟶ *ψ*-tropine + benzoic acid. H_2O_2 ⟶ *N*-oxide. Resorcinol + conc. H_2SO_4 ⟶ yellow ⟶ violet ⟶ red col. 2-Naphthol + conc. H_2SO_4 ⟶ grey ⟶ dark blue col. Produces paralysis of central nervous system. Poisonous. Exhibits local anæsthetic properties.

B,HCl: plates from EtOH. M.p. 283°.

B,HAuCl₄: yellow needles from H_2O. M.p. 208°.

B₂,H₂PtCl₆: yellow needles. M.p. 276–8°.

d-*Camphor-β-sulphonic acid salt*: prisms from EtOH–AcOEt. M.p. 176–7°. $[\alpha]_D$ + 11·1° in H_2O.

*α-Bromo-*d-*camphor-π-sulphonic acid salt*: prisms from EtOH, m.p. 190°; needles + $3H_2O$ from H_2O, m.p. 73°. $[\alpha]_D$ + 47·3° in H_2O.

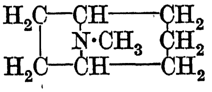
B,C₆H₅·COOH: m.p. 60–1°.

N-*Oxide*: $C_{15}H_{19}O_3N$. MW, 261. Cryst. M.p. 152–3°. *Hydrochloride*: m.p. 200°.

Picrate: needles from dil. EtOH. Darkens at 215–20°, m.p. 243–5°.

Willstätter, *Ber.*, 1896, **29**, 943.
Hesse, *Ann.*, 1892, **271**, 208.

Tropane (*Dihydrotropidine*)



$C_8H_{15}N$ MW, 125

Liq. B.p. 167°. D_4^{20} 0·931. Misc. with cold H_2O. Spar. misc. with hot H_2O. H_2O_2 ⟶ tropane-*N*-oxide. Dist. of hydrochloride in current of HCl ⟶ nortropane.

B,HAuCl$_4$: plates from EtOH. M.p. 245–6°.
B$_2$,H$_2$PtCl$_6$: orange-yellow plates from H_2O. Decomp. at 229–30°.
N-*Oxide* : $C_8H_{15}ON$. MW, 141. Hygroscopic cryst. *B$_2$,H$_2$PtCl$_6$* : orange-yellow needles. M.p. 228° decomp.
Methiodide : cryst. from H_2O. Does not melt below 300°.
Picrate : needles. M.p. 281° decomp.

Hess, *Ber.*, 1918, **51**, 1007.
Coleman, Carnes, *Chem. Abstracts*, 1943, **37**, 5703.

Tropanol.
See Tropine.
ψ-Tropanol.
See ψ-Tropine.
Tropanone.
See Tropinone.
Tropeïn.
See under Tropine.
Tropene.
See Tropidine.
Tropic Acid (1-*Phenylhydracrylic acid, β-hydroxyhydratropic acid, 2-hydroxy-1-phenylpropionic acid, α-hydroxymethylphenylacetic acid*)

C_6H_5
HO·CH_2·CH·COOH

$C_9H_{10}O_3$ MW, 166

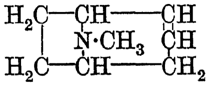
d-.
Needles, scales or prisms from H_2O, needles from C_6H_6. M.p. 129–30°. $[\alpha]_D^{16}$ + 72·2° in EtOH, $[\alpha]_D$ + 81·6° in H_2O.
Quinine salt : needles or plates from EtOH. M.p. 191·5–192·5°. Spar. sol. H_2O. $[\alpha]_D$ − 104° in EtOH.

l-.
Needles from H_2O, plates from AcOEt. M.p. 129–30°. Spar. sol. cold C_6H_6. Sol. AcOEt, methyl ethyl ketone. $[\alpha]_D^{13}$ − 72·5° in EtOH, $[\alpha]_D$ − 81·2° in H_2O.
Quinine salt : plates or needles from EtOH. M.p. 185–6°. More sol. EtOH than *d*-salt. $[\alpha]_D$ − 140·7° in EtOH.
Tropine ester : *see* Hyoscyamine.
Scopine ester : *see* Hyoscine.

dl-.
Needles or plates from H_2O, cryst. from C_6H_6. M.p. 118°. Sol. 49 parts H_2O at 14·5°. Very sol. hot H_2O. Sol. EtOH, Et_2O. Spar. sol. C_6H_6. Insol. CS_2. $k = 7{\cdot}50 \times 10^{-5}$ at 25°. Resolved into active components through quinine salt. Heat at 160° ⟶ tropide. Ox. ⟶ C_6H_5·CHO + C_6H_5·COOH. Boiling aq. $Ba(OH)_2$ or aq. KOH ⟶ atropic acid. Conc. HCl at 140° ⟶ α-isatropic acid.
Me ester : $C_{10}H_{12}O_3$. MW, 180. Needles. M.p. 36·5–37·5°. B.p. 159–62°/19 mm.
Tropine ester : *see* Atropine.
Amide : $C_9H_{11}O_2N$. MW, 165. Plates and needles from H_2O. M.p. 169·5°. Sol. hot H_2O. Spar. sol. cold H_2O.
Me ether : $C_{10}H_{12}O_3$. MW, 180. Cryst. from petrol. M.p. 62°. B.p. 134–6°/0·3 mm.
Acetyl : plates. M.p. 88–90°.

Chambon, *Compt. rend.*, 1928, **186**, 1630.
King, Palmer, *J. Chem. Soc.*, 1922, **121**, 2577.
McKenzie, Wood, *J. Chem. Soc.*, 1919, **115**, 838.
Wislicenus, Bilhuber, *Ber.*, 1918, **51**, 1237.
Natarajan, Swaminathan, *J. Am. Chem. Soc.*, 1947, **69**, 2560.

Tropidine (2-*Tropene*)

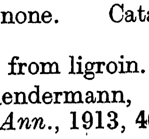


$C_8H_{13}N$ MW, 123

Liq. B.p. 163°. Sol. EtOH. Et_2O. Spar. misc. with hot H_2O. Mod. misc. with cold H_2O. D_4^{20} 0·953. n_α^{19} 1·4884. Volatile in steam. H_2O_2 ⟶ *N*-oxide. Red. ⟶ tropane. Stable to hot H_2SO_4 and alkalis.
B,HAuCl$_4$: m.p. 212°.
B$_2$,H$_2$PtCl$_6$: orange-red cryst. Decomp. at 217°.
N-*Oxide* : $C_8H_{13}ON$. MW, 139. Hygroscopic cryst. *B$_2$,H$_2$PtCl$_6$* : orange-yellow plates from H_2O. M.p. about 220° decomp.
Methiodide : cryst. from H_2O or EtOH. M.p. about 300°.
Picrate : yellow needles from H_2O. M.p. about 285° decomp.

Willstätter, *Ber.*, 1901, **34**, 142.

Tropigenin.
See Nortropine.
Tropilene (*Cycloheptenone*-3)

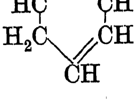


$C_7H_{10}O$ MW, 110

Liq. B.p. 186–8°. Insol. H_2O. D^0 1·0091. Reduces $NH_3.AgNO_3$ and Fehling's. Ox. ⟶ adipic and oxalic acids. Zn + AcOH ⟶ methylcyclohexanone. Catalytic red. ⟶ suberone.
Oxime : cryst. from ligroin. M.p. 80–8°.

Kötz, Blendermann, Mähnert, Rosenbusch, *Ann.*, 1913, **400**, 80.

Tropilidene ($\Delta^{1:3:5}$-*Cycloheptatriene*)

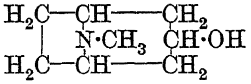


C_7H_8 MW, 92

Liq. B.p. 117°/749 mm. $D_4^{18.5}$ 0·8875. $n_\alpha^{17.5}$ 1·51751. Resinifies on standing in air. Alc. sol. + conc. H_2SO_4 ⟶ dark brownish-red col.

Willstätter, *Ann.*, 1901, **317**, 204, 269.

Tropine (3-*Tropanol*)



$C_8H_{15}ON$ MW, 141

Hygroscopic plates from Et_2O. M.p. 63°. B.p. 229°. Very sol. H_2O, EtOH. Difficultly volatile in steam. $k = 2{\cdot}74 \times 10^{-4}$ at 25°. NaOEt ⟶ ψ-tropine. H_2O_2 ⟶ *N*-oxide. CrO_3 ⟶ tropinone. $KMnO_4$ in alk. sol. ⟶ nortropine. Acid $KMnO_4$ ⟶ tropinone. H_2SO_4 + AcOH ⟶ tropidine. Soda-lime dist. ⟶ tropilidine. HI + PH_4I ⟶ tropane.

B,HAuCl₄: yellow plates. M.p. 210–12° decomp.

B_2,H_2PtCl_6: yellowish-red plates from H_2O. M.p. 197–8° decomp.

d-*Camphor*-β-*sulphonic acid salt*: plates from EtOH–AcOEt or $CHCl_3$. M.p. 236°. $[\alpha]_D$ + 32·1° in $CHCl_3$.

Picrate: rhombohedra from H_2O. M.p. 290–5°.

Me ether: $C_9H_{17}ON$. MW, 155. Cryst. powder. Does not melt below 300°. Very sol. H_2O, hot EtOH. Spar. sol. Et_2O.

Acetyl: acetyltropeïn. Liq. B.p. 235–7°.

Benzoyl: benzoyltropeïn. Plates + $2H_2O$, m.p. 56°: anhyd., cryst. from Et_2O, m.p. 41–2°. B.p. 175–80°. *B,HCl*: prisms from EtOH. M.p. 275°.

N-*Oxide*: $C_8H_{15}O_2N$. MW, 157. M.p. 238°.

Mandelic ester: *see* Homatropine.

l-*Tropic ester*: *see* Hyoscyamine.

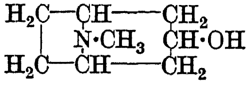
dl-*Tropic ester*: *see* Atropine.

Phenylcarbamic ester: *see* Uretropine.

Veratric ester: *see* Convolvamine.

Willstätter, *Ber.*, 1902, **35**, 1870.

ψ-Tropine (ψ-3-*Tropanol*)



$C_8H_{15}ON$ MW, 141

Stereoisomer of tropine. Prisms from C_6H_6–pet. ether or ligroin. M.p. 108–9°. B.p. 240–1°. Very sol. H_2O, EtOH, $CHCl_3$, C_6H_6. Sol. Et_2O. Ox. ⟶ tropinone. Dil. alk. $KMnO_4$ ⟶ nor-ψ-tropine. H_2O_2 ⟶ *N*-oxide. AcOH + H_2SO_4 ⟶ tropidine.

B,HCl: needles from EtOH. Sinters at 250°. M.p. 280–2° decomp.

B,HAuCl₄: yellow plates or needles from H_2O. M.p. 225° decomp.

B_2,H_2PtCl_6: orange-red plates + $4H_2O$ from H_2O. M.p. anhyd. 206°.

d-*Camphor*-β-*sulphonic acid salt*: prisms from EtOH–AcOEt. M.p. 224–6°. $[\alpha]_D$ + 26·3° in EtOH.

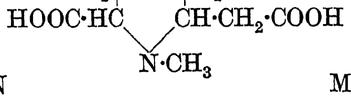
Picrate: needles or prisms from H_2O. Darkens at 245°, m.p. 258–9° decomp.

Benzoyl: *see* Tropacocaine.

N-*Oxide*: $C_8H_{15}O_2N$. MW, 157. M.p. 229°. *Hydrochloride*: m.p. 286°. *Picrate*: m.p. 257° decomp.

Willstätter, *Ber.*, 1901, **34**, 3165.

Tropinic Acid (N-*Methylpyrrolidine*-5-*carboxylic acid*-2-*acetic acid*)



$C_8H_{13}O_4N$ MW, 187

d-.

Cryst. from H_2O or EtOH.Aq. M.p. (rapid heat.) 253° decomp., (slow heat.) 247–8°. Sol. cold H_2O. Spar. sol. EtOH. Insol. Et_2O, C_6H_6. $[\alpha]_D$ + 14·8° in H_2O. Decolourises $KMnO_4$ sol. in cold.

Di-Me ester: $C_{10}H_{17}O_4N$. MW, 215. Oil. *Picrate*: needles. M.p. 120–1°. *Methiodide*: plates and needles from MeOH. M.p. 176–7° decomp.

l-.

Cryst. from H_2O. M.p. 243°. $[\alpha]_D^{20}$ − 14·8° in H_2O.

dl-.

Needles from EtOH.Aq. M.p. about 248° decomp. Sol. H_2O. Spar. sol. EtOH. Insol. Et_2O, C_6H_6. Resolved through cinchonine salt. Loses CO_2 on heating above m.p. Decolourises $KMnO_4$ sol. in cold. CrO_3 + H_2SO_4 ⟶ *N*-methylsuccinimide. HI(+ P) ⟶ *N*-methylpyrrolidine.

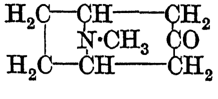
B_2,H_2PtCl_6: orange-yellow cryst. Decomp. at 100–10°.

Di-Me ester: $C_{10}H_{17}O_4N$. MW, 215. Oil. B.p. 268–72° (part. decomp.). *Picrate*: orange-yellow prisms from EtOH. M.p. 121°. *Methiodide*: prisms + $\frac{1}{2}H_2O$ from H_2O or EtOH.Aq., anhyd. plates from EtOH–Et_2O. M.p. 171–2° decomp.

Di-Et ester: $C_{12}H_{21}O_4N$. MW, 243. Oil. B.p. 160°/18·5 mm.

Willstätter, *Ber.*, 1898, **31**, 1547.
Liebermann, *Ber.*, 1891, **24**, 2587.

Tropinone (3-*Tropanone*)



$C_8H_{13}ON$ MW, 139

Needles from pet. ether. M.p. 42°. B.p. 224–5°, 125°/40 mm., 113°/25 mm. Very sol. most org. solvents. Reacts alkaline. Reduces warm $AgNO_3$. H_2O_2 ⟶ *N*-oxide.

B,HCl : prisms from EtOH. M.p. about 188–9° decomp.

B,HAuCl$_4$: yellow prisms from dil. HCl. M.p. about 163° decomp.

B$_2$,H$_2$PtCl$_6$: orange-red prisms. M.p. 191–2° decomp.

d-*Camphor-β-sulphonic acid salt* : cryst. from AcOEt. M.p. 216° decomp.

Picrate : yellow needles from H_2O. M.p. 217–18°.

N-*Oxide* : $C_8H_{13}O_2N$. MW, 155. M.p. 98°.

Oxime : prisms from ligroin. M.p. 115–16°. Very sol. H_2O, EtOH, warm Me_2CO, AcOEt, $CHCl_3$, C_6H_6. *B,HCl* : prisms from EtOH. M.p. 242° decomp. *Methiodide* : prisms from EtOH.Aq. M.p. 236° decomp.

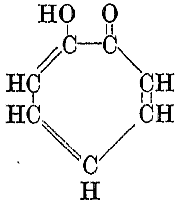
Semicarbazone : plates from EtOH. M.p. 212–13°.

Methiodide : cryst. from H_2O. M.p. 273–5°.

Dibenzylidene deriv. : yellow prisms. M.p. 152°.

Schöpf, Lehmann, *Ann.*, 1935, **518**, 1.
Willstätter, Bommer, *Ann.*, 1926, **422**, 30.
Robinson, *J. Chem. Soc.*, 1917, **111**, 766.
Willstätter, *Ber.*, 1896, **29**, 396, 947; 1898, **31**, 1540; 1900, **33**, 1169.
Karrer, Alagil, *Helv. Chim. Acta*, 1947, **30**, 1776.

Tropolone



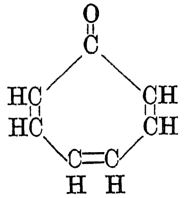
$C_7H_6O_2$ MW, 122

Needles from hexane. M.p. 48°. Sublimes. Sol. H_2O and most org. solvents. $FeCl_3$ ⟶ deep green col.

Cu deriv. : green cryst. M.p. 320° decomp. Sol. $CHCl_3$.

Dewar, *Nature*, 1945, **155**, 50.
Doering, Knox, *J. Am. Chem. Soc.*, 1950, **72**, 2305.
Haworth, Hobson, *Chemistry and Industry*, 1950, 441.
Cook, Gibb, Raphael, Somerville, *Chemistry and Industry*, 1950, 427.
Knight, Cram, *J. Am. Chem. Soc.*, 1951, **73**, 4136.

Tropone



C_7H_6O MW, 106

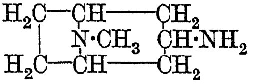
Viscous, hygroscopic oil. M.p. − 8° to − 5°. B.p. 113°/15 mm., 104–105·5°/10 mm. D_4^{22} 1·095. n_D^{22} 1·6172, n_D^{25} 1·6070.

Picrylsulphonate : m.p. 266–7°.

Picrate : m.p. 100–101°.

Dauben, Ringold, *J. Am. Chem. Soc.*, 1951, **73**, 876.
Doering, Detert, *ibid*, 876.

Tropylamine (3-*Aminotropane*)



$C_8H_{16}N_2$ MW, 140

Liq. B.p. 211°, 91–2°/12 mm. Hot Na amylate ⟶ ψ-tropylamine.

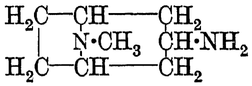
B,2HAuCl$_4$: prisms and plates. M.p. 220–1° decomp.

B,H$_2$PtCl$_6$: red plates from H_2O. M.p. 257° decomp.

Picrate : plates. M.p. 235° decomp.

Willstätter, Müller, *Ber.*, 1898, **31**, 1211.

ψ-Tropylamine (ψ-3-*Aminotropane*)



$C_8H_{16}N_2$ MW, 140

Stereoisomer of tropylamine. Liq. B.p. 213°, 107°/26 mm., 98–100°/17·5 mm. Sol. H_2O with heat evolution. Sol. EtOH, Et_2O. Strong base. Absorbs CO_2 from the air. Stable to hot Na amylate.

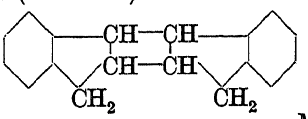
B,2HAuCl$_4$: plates, needles or prisms, with or without H_2O from H_2O. M.p. 223–4° decomp.

B,H$_2$PtCl$_6$: orange-yellow plates + $2H_2O$. M.p. 257° decomp.

Picrate : yellow cryst. M.p. 236–8° decomp.

Willstätter, Müller, *Ber.*, 1898, **31**, 1208.

Truxane (*Bisindene*)



$C_{18}H_{16}$ MW, 232

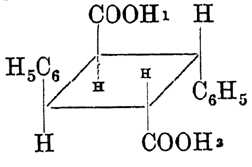
Plates. M.p. 116°. Dist. ⟶ indene. Dist. undecomp. at low pressures.

Stobbe, Zschoch, *Ber.*, 1927, **60**, 462.

Truxillamic Acid.
See under Truxillic Acid.

Truxillanilic Acid.
See under Truxillic Acid.

α-Truxillic Acid (2 : 4-*Diphenylcyclobutane*-1 : 3-*dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

Needles from EtOH.Aq, cryst. + 2MeOH from MeOH. M.p. 285°. Very sol. hot EtOH. Sol. hot AcOH. Spar. sol. Et_2O, Me_2CO, CS_2, C_6H_6, hot H_2O. Sublimes in high vacuum. $k = 4{\cdot}97 \times 10^{-5}$ at 25°. Heat of comb. C_v 2084·8 Cal., C_p 2086 Cal. KOH fusion ⟶ ε-truxillic acid. Dist. ⟶ cinnamic acid. Ac_2O at 210° ⟶ γ-truxillic anhydride. Fuming H_2SO_4 ⟶ truxone. Forms very insol. Ag salt.

Mono-Me ester: $C_{19}H_{18}O_4$. MW, 310. Needles. M.p. 195°. Sol. C_6H_6, cold Na_2CO_3.

Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Plates or needles from MeOH. M.p. 174°. B.p. about 330°. Spar. sol. cold MeOH, AcOH.

Mono-Et ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. from EtOH. M.p. 171°.

Di-Et ester: $C_{22}H_{24}O_4$. MW, 352. Needles. M.p. 146°. Spar. sol. EtOH.

Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Prisms + $\frac{1}{2}C_6H_6$ from C_6H_6–ligroin. M.p. 125°. $AlCl_3$ ⟶ truxone.

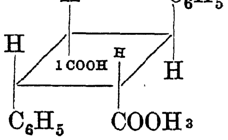
Dinitrile: $C_{18}H_{14}N_2$. MW, 258. Plates from AcOH. M.p. 233°.

1-*Amide*: α-truxillamic acid. $C_{18}H_{17}O_3N$. Needles from AcOH. M.p. 261°. Sol. EtOH, AcOH, Me_2CO. Insol. Et_2O, C_6H_6, ligroin.

3-*Anilide*: α-truxillanilic acid. $C_{24}H_{21}O_3N$. MW, 371. *d*-. Needles from EtOH.Aq. M.p. 205°. $[\alpha]_D^{20}$ + 21·8° in Me_2CO. *l*-. Needles from EtOH.Aq. M.p. 205°. $[\alpha]_D^{19}$ − 23·0° in Me_2CO. *dl*-. Needles from EtOH.Aq. M.p. 235°. Sol. EtOH, Me_2CO, AcOEt. Insol. Et_2O, C_6H_6, ligroin.

Stoermer, Stroh, *Ber.*, 1935, **68**, 2108.
Stoermer, Wegner, Carl, *Ber.*, 1923, **56**, 1683, 1690.
Stoermer, Foerster, *Ber.*, 1919, **52**, 1256.
Kohler, *Am. Chem. J.*, 1907, **28**, 238.

γ-Truxillic Acid (2 : 4-*Diphenylcyclobutane*-1 : 3-*dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

dl-.

Needles from EtOH.Aq. cryst. from AcOH. M.p. 228°. Very sol. Et_2O. Spar. sol. hot H_2O. $k = 1{\cdot}08 \times 10^{-4}$ at 25°. Heat. at 280° or HCl at 175° ⟶ α-truxillic acid. Dist. ⟶ cinnamic acid. KOH fusion ⟶ α- and ε-truxillic acids.

Mono-Me ester: $C_{19}H_{18}O_4$. MW, 310. Needles from EtOH.Aq. M.p. 183·5–184·0°. Sol. EtOH, Me_2CO, hot C_6H_6. Mod. sol. Et_2O, AcOH, C_6H_6. Insol. H_2O, ligroin, pet. ether.

Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Needles from MeOH.Aq. M.p. 126°. Sol. cold MeOH, AcOH.

Mono-Et ester: $C_{20}H_{20}O_4$. MW, 324. Needles from EtOH.Aq. M.p. 173–174·5°. Very sol. EtOH, Et_2O, AcOH, C_6H_6.

Di-Et ester: $C_{22}H_{24}O_4$. MW, 352. Needles from EtOH.Aq. M.p. 98°. Sol. EtOH, Et_2O, AcOH, C_6H_6. Spar. sol. pet. ether.

Anhydride: $C_{18}H_{14}O_3$. MW, 278. Needles from $CHCl_3$–EtOH. M.p. 191° (180–3°).

Dinitrile: $C_{18}H_{14}N_2$. MW, 258. Needles from AcOH. M.p. 204–5°.

Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Needles from C_6H_6–ligroin. M.p. 140°. $AlCl_3$ ⟶ truxone.

3-*Amide*: γ-truxillamic acid. $C_{18}H_{17}O_3N$. MW, 295. Prisms from AcOH.Aq. M.p. 240°. Insol. Et_2O, C_6H_6, pet. ether.

Imide: $C_{18}H_{15}O_2N$. MW, 277. Cryst. from AcOH or EtOH. M.p. 208°. Sol. C_6H_6. Spar. sol. Et_2O. Insol. pet. ether.

Et-imide: $C_{20}H_{19}O_2N$. MW, 305. Cryst. from EtOH.Aq. M.p. 142°.

Phenyl-imide: $C_{24}H_{19}O_2N$. MW, 353. Cryst. from AcOH.Aq. M.p. 194°.

3-*Anilide*: γ-truxillanilic acid. $C_{24}H_{21}O_3N$. MW, 371. Needles from EtOH.Aq. M.p. 228°. Sol. EtOH, Me_2CO. Insol. Et_2O, C_6H_6, ligroin.

Mono-p-*toluidide*: needles from EtOH.Aq. M.p. 268°.

Di-p-*toluidide*: needles from EtOH or AcOH. M.p. 289°.

Di-phenylhydrazide: m.p. 305°.

d-.

Mono-Me ester: cryst. from AcOH. M.p. 145·5–147°. $[\alpha]_D^{18}$ + 6·48° in Me_2CO.

Mono-Et ester: cryst. from AcOH. M.p. 142·5–143°. $[\alpha]_D^{19}$ + 19·14° in Me_2CO.

3-*Amide*: cryst. from EtOH. M.p. 258–9°.

3-*Anilide*: needles from Et_2O-ligroin. M.p. 228°. Sol. Et_2O, $CHCl_3$. Insol. C_6H_6, ligroin. $[\alpha]_D^{21}$ + 48·7° in Me_2CO.

l-.

Mono-Me ester: needles from EtOH.Aq. M.p. 145·5–147°.

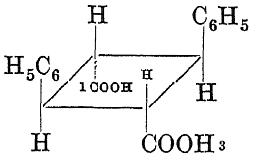
Mono-Et ester: cryst. from EtOH.Aq. M.p. 142·5–143°. $[\alpha]_D^{19}$ − 16·48° in Me_2CO.

3-*Amide*: leaflets from AcOH.Aq. M.p. 258–9°. $[\alpha]_D^{20}$ − 11·05° in AcOH.

3-*Anilide*: needles from Et_2O–ligroin. M.p. 228°. $[\alpha]_D^{23}$ − 49·54° in Me_2CO.

Stoermer, Cruse, *Ber.*, 1935, **68**, 2120.
Schenk, *Ber.*, 1930, **63**, 2706.
Stoermer, Fretwurst, *Ber.*, 1925, **58**, 2718.
Stoermer, Wegner, Carl, *Ber.*, 1923, **56**, 1683.
Stoermer, Emmel, *Ber.*, 1920, **53**, 497.
Lie, *Ber.*, 1889, **22**, 126.
Bernstein, Wallis, *J. Org. Chem.*, 1942, **7**, 261.

ε-Truxillic Acid (2 : 4-*Diphenylcyclobutane*-1 : 3-*dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

Needles from EtOH.Aq. or Et_2O. M.p. 192°. Sol. EtOH, Et_2O, $CHCl_3$. Insol. ligroin. Ba and Ca salts more sol. in cold than in hot H_2O.

Di-NH_4 salt: cryst. Decomp. at 186°.

Mono-Me ester: $C_{19}H_{18}O_4$. MW, 310. Prisms from MeOH. M.p. 131°.

Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Prisms from EtOH.Aq. M.p. 64°.

Di-Et ester: $C_{22}H_{24}O_4$. MW, 352. Prisms from EtOH. M.p. 34°.

Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Prisms from ligroin. M.p. 106–7°.

Dinitrile: $C_{18}H_{14}N_2$. MW, 258. Cryst. from EtOH. M.p. 224°.

Imide: $C_{18}H_{15}O_2N$. MW, 277. Needles from EtOH.Aq. M.p. 198°. Sol. Et_2O, EtOH, Me_2CO, AcOH. Insol. C_6H_6, ligroin. *K salt*: cryst. M.p. 224°.

Et-imide: $C_{20}H_{19}O_2N$. MW, 305. Needles from EtOH. M.p. 144°.

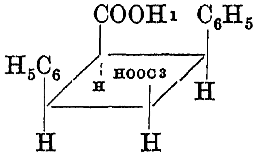
Phenylimide: $C_{24}H_{19}O_2N$. MW, 353. Needles from AcOH. M.p. 252°.

3-*Amide*: ε-truxillamic acid. $C_{18}H_{17}O_3N$. MW, 295. Needles from EtOH. M.p. 213°. Sol. EtOH, AcOH, hot H_2O. Insol. C_6H_6, ligroin.

3-*Anilide*: ε-truxillanilic acid. $C_{24}H_{21}O_3N$. MW, 371. Needles from EtOH. M.p. 239°.

Stoermer, Cruse, *Ber.*, 1935, **68**, 2121.
Stoermer, Neumaerker, Schmidt, *Ber.*, 1925, **58**, 2707, 2715.
Stoermer, Emmel, *Ber.*, 1920, **53**, 497.

η-Truxillic Acid (peri-*Truxillic acid*, 2 : 4-*diphenylcyclobutane*-1 : 3-*dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

Cryst. from AcOH–C_6H_6. M.p. 266° (287°) decomp. Sol. EtOH. Insol. Et_2O, C_6H_6. KOH fusion ⟶ ε-truxillic acid.

Mono-Me ester: $C_{19}H_{18}O_4$. MW, 310. Cryst. from MeOH. M.p. 192°.

Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. from MeOH. M.p. 104–5°.

Anhydride: $C_{18}H_{14}O_3$. MW, 278. Prisms from AcOH. M.p. 285°.

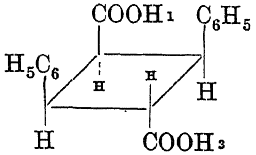
1-*Amide*: η-truxillamic acid. $C_{18}H_{17}O_3N$. MW, 295. Prisms from AcOH–C_6H_6. M.p. 256·5°. Spar. sol. AcOH. Insol. EtOH.

Imide: $C_{18}H_{15}O_2N$. MW, 277. Cryst. M.p. 237°.

1-*Anilide*: η-truxillanilic acid. $C_{24}H_{21}O_3N$. MW, 371. Cryst. from EtOH. M.p. 247°.

Stoermer, Möller, *Ber.*, 1935, **68**, 2131, 2132.
Stoermer, Bachér, *Ber.*, 1924, **57**, 15.

***epi*-Truxillic Acid** (2 : 4-*Diphenylcyclobutane*-1 : 3-*dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

Cryst. from EtOH.Aq. or C_6H_6–AcOH. M.p. 285–7°. Very spar. sol. Et_2O, C_6H_6. Heat at m.p. ⟶ ε-truxillic acid. Ac_2O ⟶ ε-acid. KOH fusion ⟶ ε-acid.

1-*Mono-Me ester*: $C_{19}H_{18}O_4$. MW, 310. Cryst. from MeOH. M.p. 204·5°.

3-*Mono-Me ester*: prisms from MeOH. M.p. 141°.

Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. M.p. 111–12°.

Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Needles from ligroin. M.p. 128–9°.

3-*Amide*: *epi*-truxillamic acid. $C_{18}H_{17}O_3N$. MW, 295. Cryst. M.p. 263°.

Diamide: $C_{18}H_{18}O_2N_2$. MW, 294. Needles from EtOH.Aq. M.p. 252–3°.

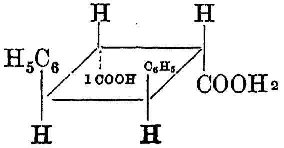
Dinitrile: $C_{18}H_{14}N_2$. MW, 258. Needles from EtOH. M.p. 167°.

Stoermer, Möller, *Ber.*, 1935, **68**, 2132.
Stoermer, Bachér, *Ber.*, 1924, **57**, 15.

Truxinamic Acid.
See under Truxinic Acid.

Truxinanilic Acid.
See under Truxinic Acid.

β-Truxinic Acid (3 : 4-*Diphenylcyclobutane*-1 : 2-*dicarboxylic acid, isotruxillic acid*)



$C_{18}H_{16}O_4$ MW, 296

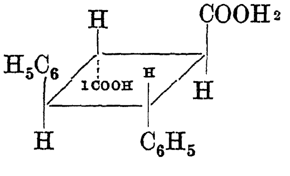
Cryst. from AcOH. M.p. 209–209·5°. Mod. sol. hot H_2O. KOH fusion ⟶ δ-truxinic acid. Dist. ⟶ cinnamic acid + stilbene. Ox. ⟶ benzil + benzoic acid. Readily forms anhydride. Forms cryst. insol. Ca and Ba salts.

Di-NH_4 salt: cryst. M.p. 187°. Sol. hot EtOH.

Mono-Me ester: $C_{19}H_{18}O_4$. MW, 310. Cryst. from C_6H_6. M.p. 164°.
Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Plates from Et_2O. M.p. 76°. Very sol. EtOH, Et_2O. Heat. of comb. C_p. 2422·9 Cal., C_v 2421·1 Cal.
Mono-Et ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. from C_6H_6. M.p. 133°. Sol. EtOH, Et_2O. Spar. sol. C_6H_6. Insol. ligroin.
Di-Et ester: $C_{22}H_{24}O_4$. MW, 352. Cryst. M.p. 49–50°.
*Mono-*d*-menthyl ester*: $C_{28}H_{34}O_4$. MW, 434. Cryst. from EtOH. M.p. 208°. Sol. Me_2CO, Et_2O, warm C_6H_6. Spar. sol. hot ligroin. $[\alpha]_D^{20}$ − 20·9° in Me_2CO.
*Mono-*l*-menthyl ester*: needles from EtOH. M.p. 149°. Very sol. Et_2O, EtOH, Me_2CO, C_6H_6. Sol. hot ligroin. $[\alpha]_D^{20}$ − 37·3° in Me_2CO.
Anhydride: $C_{18}H_{14}O_3$. MW, 278. Prisms from C_6H_6. M.p. 116°.
Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Plates from C_6H_6–pet. ether. M.p. 96°. Very sol. Et_2O, $CHCl_3$, C_6H_6. Spar. sol. pet. ether.
2-*Amide*: β-truxinamic acid. $C_{18}H_{17}O_3N$. MW, 295. Cryst. from EtOH.Aq. M.p. 194°. Sol. EtOH, Me_2CO. Insol. Et_2O, C_6H_6, ligroin.
Imide: $C_{18}H_{15}O_2N$. MW, 277. Prisms from EtOH. M.p. 224–5°. Sol. AcOH.
Phenylimide: $C_{24}H_{19}O_2N$. MW, 353. Cryst. M.p. 184°.
2-*Anilide*: β-truxinanilic acid. $C_{24}H_{21}O_3N$. MW, 371. *dl*-. Cryst. from Me_2CO.Aq. M.p. 210°. d-*Menthyl ester*: m.p. 171°. $[\alpha]_D^{20}$ − 63° in Me_2CO.

Stoermer, Lachmann, *Ber.*, 1926, **59**, 642.
Stoermer, Laage, *Ber.*, 1921, **54**, 96.
Stobbe, *Ber.*, 1919, **52**, 666.
Stoermer, Starck, Anker, *Ber.*, 1937, **70**, 483.

δ-Truxinic Acid (3 : 4-*Diphenylcyclobutane-1 : 2-dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

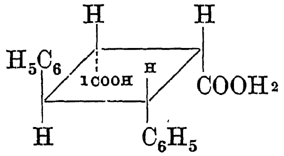
dl-.
Cryst. from C_6H_6–AcOH, needles from hot H_2O, plates + $1C_6H_6$ from C_6H_6. M.p. 175°. Very sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. boil. H_2O, C_6H_6.
NH_4 salt: prisms. M.p. 206–8° decomp.
Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Needles from MeOH.Aq. M.p. 77°. Sol. EtOH, C_6H_6, ligroin.
Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Cryst. from C_6H_6–ligroin. M.p. 78°. Unstable in air. Sol. Et_2O, $CHCl_3$, CS_2, C_6H_6. Spar. sol. ligroin.
2-*Amide*: δ-truxinamic acid. $C_{18}H_{17}O_3N$. MW, 295. M.p. 189°.
2-*Anilide*: δ-truxinanilic acid. $C_{24}H_{21}O_3N$. MW, 371. M.p. 225°.

d-.
Cryst. M.p. 157–8°. $[\alpha]_D^{20}$ + 8·06° in Me_2CO.
Quinine salt: needles from EtOH.Aq. M.p. 135° decomp.

l-.
Needles. M.p. 158–9°. $[\alpha]_D^{20}$ − 8·3° in Me_2CO.
Cinchonine salt: cryst. from EtOH.Aq. M.p. 192°.
Di-Me ester: cryst. from MeOH. M.p. 45°. $[\alpha]_D^{20}$ − 11·1° in Me_2CO.
2-*Amide*: m.p. 206°.

Stoermer, Bachér, *Ber.*, 1922, **55**, 1860.
Stoermer, Klockmann, *Ber.*, 1925, **58**, 1164.
Stoermer, Starck, Anker, *Ber.*, 1937, **70**, 483.

ζ-Truxinic Acid (3 : 4-*Diphenylcyclobutane-1 : 2-dicarboxylic acid*)



$C_{18}H_{16}O_4$ MW, 296

dl-.
Needles from Me_2CO.Aq. M.p. 239°. Sol. EtOH, Et_2O, Me_2CO. Spar. sol. AcOH, $CHCl_3$, CS_2, C_6H_6. Insol. H_2O, pet. ether. KOH fusion or hot HCl ⟶ δ-truxinic acid. Forms spar. sol. Ca and Ba salts.
NH_4 salt: needles. Decomp. at 160°.
1-*Mono-Me ester*: $C_{19}H_{18}O_4$. MW, 310. Cryst. from MeOH. M.p. 198°. Sol. EtOH, Et_2O, AcOH, C_6H_6. *Chloride*: cryst. from C_6H_6–ligroin. M.p. 104–5°.
2-*Mono-Me ester*: cryst. from MeOH. M.p. 201°. Sol. EtOH, Et_2O, AcOH, C_6H_6. *Chloride*: cryst. from C_6H_6–ligroin. M.p. 120°.
Di-Me ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. from EtOH. M.p. 116°.
Mono-Et ester: $C_{20}H_{20}O_4$. MW, 324. Cryst. from EtOH. M.p. 190°. Sol. EtOH, Et_2O, AcOH, C_6H_6.
Di-Et ester: $C_{22}H_{24}O_4$. MW, 352. Needles. M.p. 80°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$, C_6H_6.
Anhydride: $C_{18}H_{14}O_3$. MW, 278. Cryst. from C_6H_6. M.p. 150°. Sol. EtOH, Me_2CO, $CHCl_3$, C_6H_6. Spar. sol. Et_2O, pet. ether. Stable to H_2O.
Dichloride: $C_{18}H_{14}O_2Cl_2$. MW, 333. Prisms from C_6H_6–pet. ether. M.p. 150°. Sol. EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin, pet. ether.

1-*Amide*: ζ-truxinamic acid. $C_{18}H_{17}O_3N$. MW, 295. Needles from MeOH. M.p. 222°. Sol. AcOH.

2-*Amide*: needles from Me_2CO. M.p. 204°.

Imide: $C_{18}H_{15}O_2N$. MW, 277. Needles from EtOH. M.p. 168–168·5°. Sol. hot Na_2CO_3.

Phenylimide: $C_{24}H_{19}O_2N$. MW, 353. Needles from EtOH. M.p. 180°.

1-*Anilide*: ζ-truxinanilic acid. $C_{24}H_{21}O_3N$. MW, 371. Cryst. from epichlorohydrin. M.p. 237°.

2-*Anilide*: needles from EtOH.Aq. M.p. 214°.

d-.

Cryst. from Me_2CO. M.p. 222°. $[\alpha]_D^{20}$ + 65·42° in EtOH.

Di-Me ester: cryst. from MeOH. M.p. 106°. $[\alpha]_D$ + 89·88° in Me_2CO.

l-.

Cryst. from Me_2CO. M.p. 222°. $[\alpha]_D^{20}$ − 78·37° in EtOH.

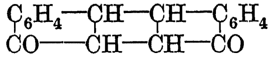
Cinchonine salt: needles from EtOH. M.p. 192°.

Anhydride: cryst. M.p. 162°. $[\alpha]_D$ − 145·84° in Me_2CO.

Dichloride: cryst. M.p. 160°. $[\alpha]_D$ − 98·6° in C_6H_6.

Stoermer, Klockmann, *Ber.*, 1925, **58**, 1164.
Stoermer, Scholtz, *Ber.*, 1921, **54**, 85.
Stoermer, Starck, Anker, *Ber.*, 1937, **70**, 483.

α-Truxone



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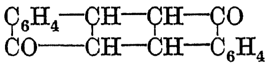


$C_{18}H_{12}O_2$ MW, 260

Plates from AcOH, powder from C_6H_6, needles from dil. HNO_3. M.p. 294°. Spar. sol. most org. solvents. Sublimes. Very stable to oxidising agents.

Dioxime: needles. Does not melt below 300° Spar. sol. most org. solvents. *Di-Me ether*: cryst. from EtOH. M.p. 214°. *Diacetyl*: needles. M.p. 261°.

Anil: needles. M.p. 270° decomp.

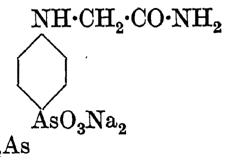
Phenylhydrazone: yellow needles. M.p. 270°.

Stobbe, Zschoch, *Ber.*, 1927, **60**, 470.
Stoermer, Foerster, *Ber.*, 1919, **52**, 1255.

Trypaflavine.

See Acriflavine.

Tryparsamide

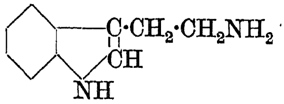


$C_8H_9O_4N_2Na_2As$ MW, 318

Cryst. + $3H_2O$. Employed in treatment of trypanosome and spirochæte infections. Low toxicity.

Jacobs, Heidelberger, *Organic Syntheses*, Collective Vol. I, 475.

Tryptamine (3-[ω-*Aminoethyl*]-*indole*, 2-[3-*indolyl*]-*ethylamine*)



$C_{10}H_{12}N_2$ MW, 160

Needles from ligroin. M.p. 146° (115°). Sol. EtOH, Me_2CO. Prac. insol. H_2O, Et_2O, $CHCl_3$, C_6H_6.

B,HCl: prisms. M.p. 248–9°.

B_2,$CH_2(COOH)_2$: needles from AcOEt. M.p. 162° decomp.

B_2,$(CH_2{\cdot}COOH)_2$: m.p. 201°.

B_2,$(CH_2{\cdot}CH_2{\cdot}COOH)_2$: needles from AcOEt. M.p. 203° decomp.

N-*Thioformyl*: plates from $CHCl_3$–pet. ether. M.p. 82°.

N-*Acetyl*: cryst. from pet. ether. M.p. 77°.

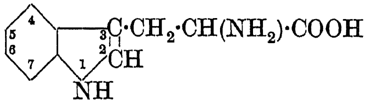
N-*Benzoyl*: prisms. M.p. 137–8°.

N-Me: *see* Dipterine.

Picrate: dark red prisms from Et_2O. M.p. 247° decomp.

Majima, Hoskins, *Ber.*, 1925, **58**, 2045.
Manske, *J. Am. Chem. Soc.*, 1929, **51**, 1202.
Späth, Lederer, *Ber.*, 1930, **63**, 123.

Tryptophan (1-*Amino*-2-[3-*indolyl*]-*propionic acid*, 2-[3-*indolyl*]-α-*alanine*)



$C_{11}H_{12}O_2N_2$ MW, 204

l-.

Constituent of many plants. Enzymatic hyd. product of most plant and animal proteins. Plates from EtOH.Aq. M.p. 278° (252°, 289°). Sol. boiling H_2O, hot EtOH. Spar. sol. cold H_2O, EtOH. Insol. $CHCl_3$. Aq. sol. shows acid reaction. $[\alpha]_D^{20}$ − 33·4° in EtOH, $[\alpha]_D^{20}$ + 6·1° in *N*/NaOH. Heat with Py or 25% HCl at 170° ⟶ *dl*-. $FeCl_3$ ⟶ indole-3-aldehyde. Br water ⟶ reddish-violet col. KOH fusion ⟶ skatole + oxalic acid + glyoxylic acid.

B,HCl: needles from MeOH. M.p. 257° decomp.

Picrate: red needles and plates. M.p. 195–6° slight decomp.

Picrolonate: orange-red needles from H_2O. M.p. 203–4° decomp.

Me ester: $C_{12}H_{14}O_2N_2$. MW, 218. Plates from Et_2O. M.p. 89·5°. Very sol. MeOH.

Sol. Et_2O, AcOEt. Spar. sol. pet. ether. *B,HCl*: microneedles from MeOH or AcOEt. M.p. 214° decomp.

Chloride: $C_{11}H_{11}ON_2Cl$. MW, 222·5. *B,HCl*: darkens at 172°, sinters at 208°, m.p. 228° decomp.

Amide: $C_{11}H_{13}ON_3$. MW, 203. M.p. 167–70°. $[\alpha]_D^{20}$ − 7·9° in EtOH.

Et-amide: $C_{13}H_{17}ON_3$. MW, 231. M.p. 67–9°. $[\alpha]_D^{20}$ − 14·4° in EtOH.

Di-Et amide: $C_{15}H_{21}ON_3$. MW, 259. M.p. 183–5°. $[\alpha]_D^{20}$ − 24·7° in EtOH.

Anilide: m.p. 83–5°. $[\alpha]_D^{20}$ − 9·5° in EtOH.

Et-anilide: m.p. 97–9°. $[\alpha]_D^{20}$ − 4·2° in EtOH.

N-*Chloroacetyl*: plates from H_2O. M.p. 159°.

N-*Phenoxyacetyl*: m.p. 196–7°. $[\alpha]_D^{20}$ + 1·5°.

N-*Benzoyl*: cryst. from H_2O. M.p. 104–5°. $[\alpha]_D^{20}$ − 37°.

N-p-*Nitrobenzoyl*: m.p. 121°.

N-*Benzenesulphonyl*: cryst. from EtOH.Aq. M.p. 185° decomp.

dl-.

Plates from EtOH.Aq. M.p. 275–82°.

α-N-*Me*: $C_{12}H_{14}O_2N_2$. MW, 218. Needles from EtOH.Aq. Darkens at 280°, m.p. 297° decomp. *Picrate*: plates from MeOH–pet. ether. M.p. 186° decomp.

α-N-*Di-Me*: $C_{13}H_{16}O_2N_2$. MW, 232. Needles from Et_2O–pet. ether. M.p. 49–50°. *Methiodide*: needles from MeOH. M.p. 210–11°. *Picrate*: prisms from MeOH. M.p. 170–1°.

N-*Acetyl*: cryst. from EtOH.Aq. M.p. 206°. *Et ester*: m.p. 133·5–135·5°.

N-*Benzoyl*: cryst. from EtOH.Aq. M.p. 188–91°.

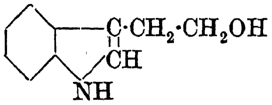
N-3 : 5-*Dinitrobenzoyl*: m.p. 240°.

N-*Benzenesulphonyl*: m.p. 185° decomp.

N-p-*Toluenesulphonyl*: needles from Me_2CO. M.p. 176°.

Boyd, Robson, *Biochem. J.*, 1935, **29**, 2256.
Gordon, Jackson, *J. Biol. Chem.*, 1935, **110**, 151.
Hoshino, Shimodaira, *Ann.*, 1935, **520**, 25.
Bauguess, Berg, *J. Biol. Chem.*, 1934, **106**, 618.
Cox, King, *Organic Syntheses*, 1930, X, 100.
Baker, Happold, *Biochem. J.*, 1940, **34**, 657.
Albertson, Archer, Suter, *J. Am. Chem. Soc.*, 1945, **67**, 37.
Spies, *J. Am. Chem. Soc.*, 1948, **70**, 3717.

Tryptophol (2-[3-*Indolyl*]-*ethyl alcohol*, 3-ω-*hydroxyethylindole*)



$C_{10}H_{11}ON$ MW, 161

Prisms from C_6H_6–pet. ether, plates from Et_2O–pet. ether. M.p. 59°. B.p. 174°/2 mm. Sol. MeOH, EtOH, Et_2O, Me_2CO, $CHCl_3$, AcOH, AcOEt. Spar. sol. cold H_2O, ligroin, pet. ether. Heat or boil. with alkalis ⟶ indole. Sol. warm conc. H_2SO_4 ⟶ red col.

N-*Benzoyl*: yellow prisms and plates from ligroin. M.p. 76°.

Picrate: red needles from H_2O. M.p. 100–1°.

Phenylurethane: m.p. 130–1°.

Jackson, *J. Biol. Chem.*, 1930, **88**, 659.
Ehrlich, *Ber.*, 1912, **45**, 884.
Bernado, Oddo, *Gazz. chim. ital.*, 1937, **69**, 10.
Snyder, Pilgrim, *J. Am. Chem. Soc.*, 1948, **70**, 3770.

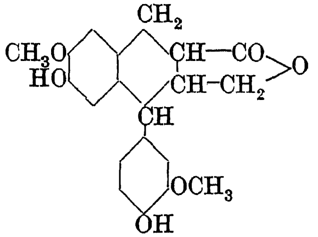
Tsuduic Acid.

See 3-Tetradecylenic Acid.

Tsugalactone.

See Tsugaresinol.

Tsugaresinol (*Tsugalactone*, *conidendrin*, *sulphite liquors lactone*)



$C_{20}H_{20}O_6$ MW, 356

Constituent of spruce resin and of the wood of *Tsuga Sieboldii*. Found in sulphite waste liquors from wood pulp manufacture. Cryst. from EtOH. M.p. 254–5°. Sol. EtOH, Et_2O, C_6H_6. Insol. pet. ether. $[\alpha]_D^{20}$ − 54·5° in Me_2CO. Alc. $FeCl_3$ ⟶ green col. Conc. H_2SO_4 + $NaNO_2$ ⟶ brown ⟶ red ⟶ blue col.

Di-Me ether: $C_{22}H_{24}O_6$. MW, 384. M.p. 178·5–179°. $[\alpha]_D^{18}$ − 99·4° in Me_2CO. *Dibromo deriv.*: m.p. 183–4°. $[\alpha]_D^{18}$ − 16·8° in Me_2CO.

Di-Et ether: $C_{24}H_{28}O_6$. MW, 412. Needles from EtOH. M.p. 178–9°.

Diacetyl: cryst. from EtOH–Me_2CO. M.p. 204–5° (222°). $[\alpha]_D^{18}$ − 68·4° in Me_2CO.

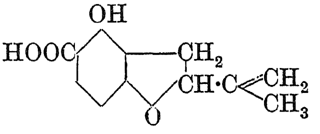
Di-p-*nitrobenzoyl*: prisms from $CHCl_3$–Et_2O. M.p. 257–8°.

Emde, Schartner, *Helv. Chim. Acta*, 1935, **18**, 344.
Haworth, Sheldrick, *J. Chem. Soc.*, 1935, 636.
Kawamura, *Chem. Zentr.*, 1932, II, 60.
Holmberg, *Ber.*, 1921, **54**, 2406.
Keimatsu, Ishiguro, Yamamoto, *J. Pharm. Soc., Japan*, 1935, **55**, 381. (*Chem. Zentr.*, 1936, I, 2568).

Tsuzuic Acid.

See 3-Tetradecylenic Acid.

Tubaic Acid



$C_{12}H_{12}O_4$ MW, 220

Needles from Et_2O-pet. ether. M.p. 129°. $[\alpha]_D^{20}$ − 73·0° in $CHCl_3$. Loses CO_2 at 185–200°. $FeCl_3$ ⟶ red col.

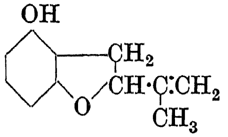
Me ester: $C_{13}H_{14}O_4$. MW, 234. Needles from MeOH.Aq. M.p. 52°. $[\alpha]_D^{19}$ − 72·8° in $CHCl_3$. $FeCl_3$ ⟶ red col.

Me ether: $C_{13}H_{14}O_4$. MW, 234. Cryst. from EtOH.Aq. M.p. 78°. *Me ester*: $C_{14}H_{16}O_4$. MW, 248. Oil. B.p. 175°/4·5 mm.

Acetyl: m.p. 133°.

Haller, LaForge, *J. Am. Chem. Soc.*, 1932, **54**, 1988; 1930, **52**, 3211.
Takei, Koide, *Ber.*, 1929, **62**, 3032.

Tubanol



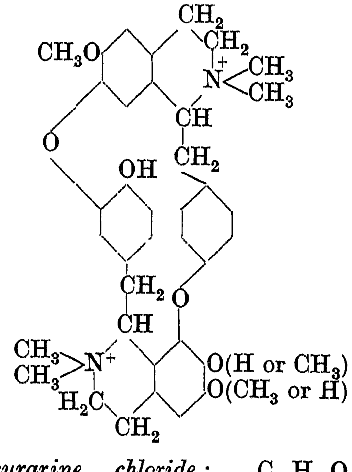
$C_{11}H_{12}O_2$ MW, 176

Viscous liq. B.p. 140°/4 mm. n_D^{20} 1·5623.

Takei, Koide, *Ber.*, 1929, **62**, 3030.
Haller, LaForge, *J. Am. Chem. Soc.*, 1930, **52**, 3207; 1931, **53**, 4460; 1932, **54**, 1988.
Haller, *J. Am. Chem. Soc.*, 1933, **55**, 3032.

Tuberculostearic Acid.
See 9-Methylstearic Acid.

Tubocurarine

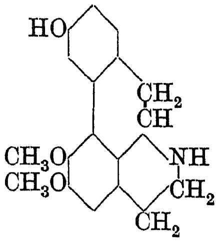


d-*Tubocurarine chloride*: $C_{38}H_{44}O_6N_2Cl_2$. MW, 695. Alkaloid from Amazon curare. M.p. 274–5° decomp. $[\alpha]_D^{22}$ + 215° in H_2O.

O-*Me-tubocurarine iodide*: m.p. 267° decomp. $[\alpha]_{5461}$ + 262° in H_2O.

King, *J. Chem. Soc.*, 1935, 1381; 1939, 1157; 1948, 1945.
Dutcher, *J. Am. Chem. Soc.*, 1946, **68**, 419.

Tuduranine



$C_{18}H_{19}O_3N$ MW, 297

Alkaloid from *Sinomenium acutum*. Poorly cryst. M.p. 105–25°. Sol. most org. solvents. Sol. alkalis. $FeCl_3$ ⟶ faint col. H·CHO + H_2SO_4 ⟶ fuchsin-red col.

B,HCl: prisms from H_2O. M.p. 286° decomp. $[\alpha]_D^{14·5}$ − 148° in MeOH.Aq.

Me ether: $C_{19}H_{21}O_3N$. MW, 311. N-*Acetyl*: prisms from EtOH. M.p. 189°. $[\alpha]_D^{18}$ − 400·17° in $CHCl_3$.

Et ether: $C_{20}H_{23}O_3N$. MW, 325. *B,HCl*: m.p. 234–6°. *B,HBr*: m.p. 246–8°. *Ethiodide*: m.p. 186–7°.

N-*Me*: $C_{19}H_{21}O_3N$. MW, 311. *Methiodide*: cryst. from EtOH. M.p. 224°.

N-*Et*-O-*Et*: $C_{22}H_{27}O_3N$. MW, 353. *Ethiodide*: M.p. 238°. $[\alpha]_D^{15}$ − 112·47° in $CHCl_3$.

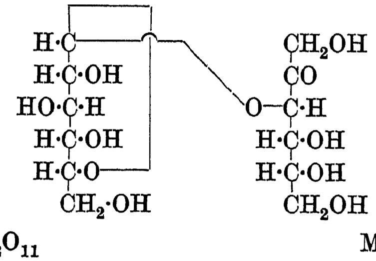
N-*Di-Et*-O-*Et*: $C_{24}H_{31}O_3N$. MW, 381. *Ethiodide*: m.p. 163–4°.

N-*Acetyl*: prisms from MeOH. M.p. 277°. $[\alpha]_D^{18}$ − 395·24° in MeOH–$CHCl_3$.

O : N-*Diacetyl*: prisms from MeOH or EtOH. M.p. 170°. $[\alpha]_D^{14}$ − 321·7° in MeOH.

Goto, *Ann.*, 1935, **521**, 175.
Goto, Shishido, *Proc. Imper. Acad., Tokyo*, 1939, **15**, 8.
Goto, Shishido, *Ann.*, 1939, **539**, 262.

Turanose (3-α-D-*Glucopyranosyl*-D-*fructose*)



$C_{12}H_{22}O_{11}$ MW, 342

Amorphous powder, or prisms + ½MeOH from MeOH. M.p. 65–70° (60–5°), MeOH free 157°. Sol. H_2O, MeOH. Less sol. EtOH. Sweet taste. $[\alpha]_D^{20}$ + 65–8° in H_2O, ($[\alpha]_D^{22}$ + 22·0° ⟶

$+ 75{\cdot}3°$ in H_2O). Reduces Fehling's. Hyd. by dil. acids or by yeast ⟶ glucose + fructose.

Phenylosazone: yellow needles from dil. EtOH. M.p. 200–5°. Sol. hot H_2O.

Phenylosotriazole deriv.: m.p. 193–4°.

Hepta-Me ether: yellow syrup. B.p. 185–90°/0·2 mm., 162–3°/0·06 mm. $[\alpha]_D^{19} + 106{\cdot}0° \longrightarrow 104{\cdot}8°$ in EtOH. n_D 1·4652. *Me glycoside*: yellow syrup. B.p. 159–62°/0·15 mm. $[\alpha]_D^{19} + 106{\cdot}7°$ in H_2O, $+ 109{\cdot}7°$ in EtOH.

Hepta-acetyl: (i) needles from Et_2O. M.p. 140–1°. $[\alpha]_D^{20} + 37°$ in $CHCl_3$. (ii) Prisms. M.p. 147°. $[\alpha]_D^{20} + 38{\cdot}7° \longrightarrow 41{\cdot}7°$ in $CHCl_3$. β-*Me glycopyranoside*: m.p. 188–9°. $[\alpha]_D^{20} + 27{\cdot}5°$ in $CHCl_3$.

α-*Turanofuranose octa-acetate*: m.p. 158°. $[\alpha]_D^{20} + 107°$ in $CHCl_3$.

β-*Turanofuranose octa-acetate*: syrup. $[\alpha]_D^{20} + 67{\cdot}4°$ in $CHCl_3$.

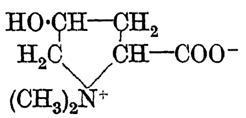
α-*Turanopyranose octa-acetate*: m.p. 194–5°. $[\alpha]_D^{20} + 103{\cdot}2°$ in $CHCl_3$.

β-*Turanopyranose octa-acetate*: m.p. 216–17°. $[\alpha]_D^{20} + 20{\cdot}5°$ in $CHCl_3$.

keto-*Turanose octa-acetate*: m.p. 96°. $[\alpha]_D^{20} + 126{\cdot}2°$ in $CHCl_3$.

Fischer, *Ber.*, 1894, **27**, 2488.
Zemplén, Braun, *Ber.*, 1926, **59**, 2230.
Zemplén, *ibid.*, 2539.
Leitch, *J. Chem. Soc.*, 1927, 588.
Aagaard, *Chem. Abstracts*, 1930, **24**, 1089.
Pacsu, *J. Am. Chem. Soc.*, 1931, **53**, 3099; 1933, **55**, 2451.
Hudson, Pacsu, *J. Am. Chem. Soc.*, 1930, **52**, 2519.
Hudson, *J. Org. Chem.*, 1944, **9**, 117, 470.
Hudson, *Advances in Carbohydrate Chemistry*, 1946, Vol. 2, 1.

Turicine



$C_7H_{13}O_3N$ MW, 159

Constituent of *Betonica wiesen* and *Stachys silvatica*. Stereoisomeric with betonicine. Needles or prisms $+ 1H_2O$ from EtOH.Aq. M.p. anhyd. 260° decomp. Very sol. H_2O. $[\alpha]_D + 41{\cdot}5°$ (anhyd.) in H_2O. Pptd. from aq. sol. by phosphotungstic acid.

B,HCl: needles or plates from EtOH. Decomp. at 224°. $[\alpha]_D + 25{\cdot}7°$ in H_2O.

B,HAuCl$_4$: yellow prisms. Decomp. at 230–2°.

B_2,H_2PtCl_6,H_2O: m.p. 223° decomp.

Goodson, Clewer, *J. Chem. Soc.*, 1919, **115**, 931.
Küng, Trier, *Z. physiol. Chem.*, 1913, **85**, 209.

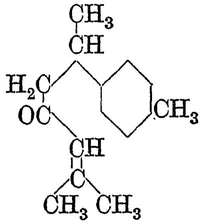
Turmerone (*Tumerone*)

$C_{15}H_{22}O$ MW, 218

Occurs in essential oil from *Curcuma longa*. Oil. B.p. 125–6°/10 mm.

Rupe, Clar, Pfau, Plattner, *Helv. Chim. Acta*, 1934, **17**, 372.

ar-**Turmerone**



$C_{15}H_{20}O$ MW, 216

Occurs in essential oil from *Curcuma longa*. Pale yellow oil. B.p. 159–60°/10 mm. D^{20} 0·9571. n_D^{20} 1·5219. $[\alpha]_D^{20} + 82{\cdot}21°$. Cyclises on warming with $AlCl_3$ in CS_2 to *cyclo-ar*-tumerone. M.p. 33°. B.p. 167°/11 mm. *Semicarbazone*: m.p. 222°. *Oxime*: m.p. 108°.

Oxime: oil. B.p. 179–80°/10 mm.

Semicarbazone: needles from C_6H_6. M.p. 106°.

2 : 4-*Dinitrophenylhydrazone*: orange-yellow needles. M.p. 133–4°.

Rupe, Gassmann, *Helv. Chim. Acta*, 1936, **19**, 569.
Rupe, Clar, Pfau, Plattner, *Helv. Chim. Acta*, 1934, **17**, 372.
Mukherji, *Chem. Abstracts*, 1943, **37**, 1409.
Colonge, Chambion, *Compt. rend.*, 1946, **222**, 557.
Mukherjee, *J. Indian Chem. Soc.*, 1947, **24**, 341.

Tutin

$C_{15}H_{18}O_6$ MW, 294

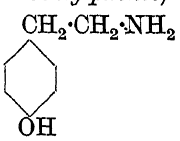
Poisonous principle of the *Coriaria* species of New Zealand. Cryst. from EtOH. M.p. 209–10°.

Acetyl deriv.: m.p. 177°.

Dihydro deriv.: m.p. 190–2°.

Slater, *J. Chem. Soc.*, 1943, 50, 143; 1949, 806.

Tyramine (*Tyrosamine*, 2-p-*hydroxyphenylethylamine*, p-β-*aminoethylphenol*)



$C_8H_{11}ON$ MW, 137

Chief pressor base found in some extracts of ergot, putrified animal tissues, mature cheese, mistletoes. Cryst. from EtOH or anisole, plates or needles from C_6H_6. M.p. 164–164·5°. B.p. 205–7°/25 mm., 195°/13 mm., 165–7°/2 mm. Spar. sol. xylene. Sol. 95 parts H_2O at 15°.

Sol. 8 parts boiling EtOH. Aq. sol. reacts alkaline. KOH fusion ⟶ *p*-hydroxybenzoic acid. Hydrochloride + KNO_3 in neutral sol. ⟶ tyrosol.

B,HCl: cryst. from EtOH–Et_2O. M.p. 269°.

B,H_2SO_4: m.p. 123–5° decomp.

Oxalate: m.p. 203–4°.

Picrate: prisms. M.p. 206°.

Me ether: $C_9H_{13}ON$. MW, 151. Oil with fish-like odour. B.p. 138–40°/20 mm., 132–4°/14 mm. Very spar. sol. H_2O. Absorbs CO_2 from the air. *B,HCl*: plates from EtOH. M.p. 271–2°. N-*Benzenesulphonyl*: cryst. from EtOH or C_6H_6–pet. ether. M.p. 79–80°.

N-*Me*: $C_9H_{13}ON$. MW, 151. Prisms from EtOH, plates from C_6H_6. M.p. 130°. B.p. 183–5°/9 mm. Spar. sol. H_2O. *B,HCl*: plates or needles from EtOH–Et_2O. M.p. 148·5°. *$B,(COOH)_2$*: needles from EtOH. M.p. 250° decomp. *B_2,H_2PtCl_6*: yellow needles. M.p. 205°. *Picrate*: m.p. 149°. *Picrolonate*: m.p. 234–5°. N-*Acetyl*: needles from H_2O, plates from EtOH or AcOEt. M.p. 142°. N-*Benzenesulphonyl*: plates from EtOH–Et_2O. M.p. 133·5°. *Dibenzoyl*: prisms from pet. ether. M.p. 99°.

N-*Di-Me*: *see* Hordenine.

N-*Et*: $C_{10}H_{15}ON$. MW, 165. Needles from EtOH. M.p. 157–8°. B.p. 185–7°/9 mm. *B,HCl*: pale red needles from EtOH–Et_2O. M.p. 184–5°. *$B,(COOH)_2$*: plates from EtOH. M.p. 245° decomp. *Picrolonate*: m.p. 216° decomp.

N-*Chloroacetyl*: cryst. M.p. 109°.

N-*Benzoyl*: plates from EtOH. M.p. 162°.

O : N-*Dibenzoyl*: needles from EtOH.Aq. M.p. 172°.

Tribenzoyl: m.p. 141°.

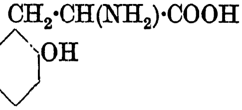
Buck, *J. Am. Chem. Soc.*, 1933, **55**, 3389.
Waser, *Helv. Chim. Acta*, 1925, **8**, 766.
Barger, *J. Chem. Soc.*, 1909, **95**, 1127, 1722.
Gryszkiewicz-Trochimowski, *Chem. Zentr.*, 1938, I, 872.
Corti, *Helv. Chim. Acta*, 1949, **32**, 681.

Tyrocidine.

The hydrochloride of a polypeptide. Isolated from the culture-medium of *Bacillus brevis*. Possesses bactericidal activity. M.p. about 240° decomp. $[\alpha]_D^{25}$ − 101° in 95% EtOH.

Hotchkiss, Dubos, *J. Biol. Chem.*, 1941, **141**, 155.
Goldstein, *Chem. Abstracts*, 1949, **43**, 3884.

***o*-Tyrosine** (2-o-*Hydroxyphenyl-α-alanine*, 1-*amino*-2-o-*hydroxyphenyl-propionic acid*, 2-*hydroxy-α-aminohydrocinnamic acid*)



$C_9H_{11}O_3N$ MW, 181

Needles from H_2O, plates from EtOH.Aq. M.p. 249–50°. Sol. 500 parts H_2O at 17°. Sol. warm AcOH. Insol. Et_2O. $FeCl_3$ ⟶ red ⟶ violet col.

B,HCl: prisms. Decomp. at 180°.

Me ester: $C_{10}H_{13}O_3N$. MW, 195. *B,HCl*: m.p. 179°.

Anhydride: *diacetyl*, needles from EtOH. M.p. 225°.

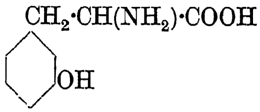
Me ether: $C_{10}H_{13}O_3N$. MW, 195. Needles from H_2O. M.p. 206° decomp.

N-*Benzoyl*: cryst. from H_2O. M.p. 176°.

Dibenzoyl: cryst. from C_6H_6. M.p. 172°.

Dickinson, Marshall, *J. Chem. Soc.*, 1929, 1497.
Ueda, *Chem. Zentr.*, 1928, I, 2618.
Johnson, Scott, *J. Am. Chem. Soc.*, 1915, **37**, 1853.

***m*-Tyrosine** (2-m-*Hydroxyphenyl-α-alanine*, 1-*amino*-2-m-*hydroxyphenyl-propionic acid*, 3-*hydroxy-α-aminohydrocinnamic acid*)



$C_9H_{11}O_3N$ MW, 181

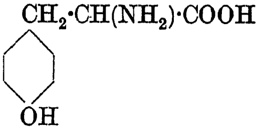
Needles or plates from H_2O, plates from EtOH.Aq. M.p. 275° (280°). Sol. 120 parts H_2O at 17°, 22 parts at 100°. Sol. cold AcOH.Aq. Spar. sol. EtOH.

Anhydride: cryst. from EtOH.Aq. M.p. 276–7°. *Diacetyl*: needles from EtOH. M.p. 189–90°.

N-*Benzoyl*: cryst. from H_2O. M.p. 180°.

Dickinson, Marshall, *J. Chem. Soc.*, 1929, 1497.
Ueda, *Chem. Zentr.*, 1928, I, 2618.
Blum, *Chem. Zentr.*, 1908, II, 1946.

Tyrosine (p-*Tyrosine*, 2-p-*hydroxyphenyl-α-alanine*, 1-*amino*-2-p-*hydroxyphenyl-propionic acid*, 4-*hydroxy-α-aminohydrocinnamic acid*)



$C_9H_{11}O_3N$ MW, 181

d-.

Cryst. Melts with decomp. $[\alpha]_D^{20}$ + 8·64° in 21% HCl. Aq. sol. + tyrosinase ⟶ red col.

Me ester: *N-benzoyl*, m.p. 150–1°.

N-*Acetyl*: m.p. 153–4°. $[\alpha]_D^{25}$ − 48·3°.

O: N-*Diacetyl*: m.p. 171–2°. $[\alpha]_D^{26}$ − 38·7°.

N-*Benzoyl*: m.p. 165·5°. $[\alpha]_D^{20}$ − 19·59° in alk. sol.

l-.

Widely distributed in plant and animal proteins. Needles from H_2O. Decomp. (slow

heat.) at 290–5°, (rapid heat.) at 314–18°. Triboluminescent. Sol. 2491 parts H_2O at 17°, 13,500 parts cold 96% EtOH, 700 parts AcOH at 16°. Sol. acids, alkalis, ammonia. Insol. Et_2O. $[\alpha]_D^{20}$ − 8·07° in 21% HCl, − 9·01° in 11·6% KOH. Heat of comb. C_p 1071·2 Cal., C_v 1070·8 Cal. $k = 4 \times 10^{-9}$ at 25°. Heat at 270° ⟶ tyramine. Stable to saturated HCl and HBr at 240°. NaOH fusion ⟶ *p*-hydroxybenzoic acid. Heat with $Ba(OH)_2$.Aq. at 170° ⟶ *dl*-form. Boiling dil. AcOH sol. + $NaNO_2$ ⟶ violet or red col. HCl sol. + Cl water + NH_3 ⟶ red col. Tyrosinase ⟶ red ⟶ black "melanin" pigment. Warm HNO_3 ⟶ yellow col. TiO_2 in H_2SO_4 ⟶ dark orange-yellow col. Paraformaldehyde + pure conc. H_2SO_4 ⟶ green col. Alloxan in H_2O ⟶ murexide col. Forms cryst. salts. Ba and Ag salts spar. sol. cold H_2O.

Cu salt: dark blue needles. Spar. sol. cold H_2O. Sol. hot H_2O.

Picrolonate: decomp. at 260°.

Me ester: $C_{10}H_{13}O_3N$. MW, 195. Prisms from AcOEt. M.p. 135–6°. Very sol. MeOH. Sol. hot H_2O, EtOH, AcOEt, alkalis. Spar. sol. cold H_2O, Et_2O, C_6H_6. $[\alpha]_D^{20}$ + 25·75° in MeOH. N-*Me*: $C_{11}H_{15}O_3N$. MW, 209. Prisms from AcOEt. M.p. 116–17°. Spar. sol. Me_2CO.

Et ester: $C_{11}H_{15}O_3N$. MW, 209. Prisms from AcOEt. M.p. 108–9°. Sol. EtOH, hot C_6H_6, AcOEt. Spar. sol. cold H_2O, Et_2O. $[\alpha]_D^{20}$ + 20·4° in EtOH. *Hydrochloride*: needles from EtOH–Et_2O or AcOEt. M.p. 166°. N-*Chloroacetyl*: needles from $CHCl_3$–pet. ether. M.p. 88–9°. N-*Bromoacetyl*: m.p. 101°. N-*Iodoacetyl*: plates from C_6H_6. M.p. 120°. N-*Benzoyl*: $[\alpha]_D^{20}$ − 23·2°. N-p-*Toluenesulphonyl*: needles from $CHCl_3$–pet. ether. M.p. 114°.

Amide: $C_9H_{12}O_2N_2$. MW, 180. Prisms or plates from EtOH. M.p. 153–4°. Sol. H_2O, EtOH. $[\alpha]_D^{20}$ + 19·47° in H_2O.

Anhydride: $(C_9H_9O_2N)_n$. Two forms. (α-) Needles from EtOH. M.p. 278–9°. Spar. sol. cold H_2O, cold EtOH. Cryst. from EtOH repeatedly ⟶ β-. (β-) Greyish powder. M.p. 279° slight decomp.

Hydrazide: m.p. 195·5°.

N-*Me*: *see* Surinamine.

Me ether: plates. M.p. 264–5°. $[\alpha]_{5461}^{29}$ − 5·9° in HCl. *B,H_2SO_4*: m.p. 191°. *B,HCl*: needles from dil. HCl. M.p. 237–8° decomp. *Picrolonate*: needles. Softens at 145°, m.p. 174° decomp. N-*Acetyl*: plates from H_2O. M.p. 150–1°. N-*Benzoyl*: needles from H_2O. M.p. 136–7°.

N-*Formyl*: prisms or plates + $1H_2O$ from H_2O. Anhyd. in vacuo at 100°. M.p. anhyd. 171–4° decomp. $[\alpha]_D^{20}$ + 84·8° in EtOH.

N-*Acetyl*: cryst. from H_2O. Plates from dioxan. M.p. 153–4°. $[\alpha]_D^{26}$ + 47·3°.

O-*Acetyl*: m.p. 213–14° decomp.

N-*Chloroacetyl*: prisms from H_2O. M.p. 155–6°.

N-*Benzoyl*: plates from H_2O. M.p. 165–6°.

N-p-*Toluenesulphonyl*: needles or prisms from EtOH.Aq. M.p. 187–8°.

O : N-*Diacetyl*: cryst. M.p. 172°.

O : N-*Dibenzoyl*: needles from AcOH. M.p. 211–12°.

dl-.

Occurs naturally. Plates or needles from H_2O. Decomp. (slow heat.) at 290–5°, (rapid heat.) at 340°. Sol. 2454 parts H_2O at 20°, 154 at 100°. Insol. cold EtOH, Et_2O.

Et ester: *B,HCl*: m.p. 166°. O : N-*Diacetyl*: m.p. 90°. N-*Benzoyl*: needles from EtOH.Aq. or C_6H_6–ligroin. M.p. 122–3°.

Isoamyl ester: $C_{14}H_{21}O_3N$. MW, 251. Yellow needles. M.p. 68–70°. Sol. EtOH, AcOEt, $CHCl_3$, C_6H_6. Spar. sol. H_2O. *B,HCl*: needles from AcOEt–Et_2O. M.p. 181–2°. N-*Benzoyl*: needles. M.p. 106–7°.

Amide: N-*benzoyl*, needles from EtOH.Aq. M.p. 232–3°.

Anhydride: $(C_9H_9O_2N)_n$. Two forms. (α-) Needles from EtOH. M.p. 278–9°. (β-) Amorph. M.p. 279°.

Hydrazide: needles from EtOH. M.p. 171°. N-*Benzoyl*: needles. M.p. 229–30°.

Me ether: plates or prisms. M.p. about 295° decomp. Sol. HCl, NH_3. Spar. sol. H_2O, hot AcOH.

N-*Me*: needles. Decomp. at 318°.

O : N-*Di-Me*: $C_{11}H_{15}O_3N$. MW, 209. Needles from H_2O. Decomp. at 220–55°.

p-*Nitrobenzyl ether*: cryst. from EtOH. M.p. 209–11°. N-*Acetyl*: m.p. 146·7°.

N-*Acetyl*: monohydrate. M.p. 94–5°.

O : N-*Diacetyl*: cryst. M.p. 168–70°.

N-*Benzoyl*: needles from AcOH or hot H_2O. M.p. 191–3°. *Anilide*: microcryst. M.p. 212°.

p-*Toluenesulphonyl*: columns. M.p. 224–6°.

Bucherer, Lieb, *J. prakt. Chem.*, 1934, **141**, 37.

Chikano, *Z. physiol. Chem.*, 1929, **180**, 249.

Curtius, Donselt, *J. prakt. Chem.*, 1917, **95**, 349.

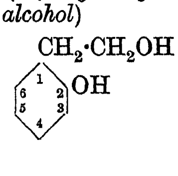
Mörner, *Z. physiol. Chem.*, 1913, **88**, 126.

Fischer, *Ber.*, 1901, **34**, 451; 1899, **32**, 3644.

Sealock, *J. Biol. Chem.*, 1946, **166**, 1.

Borrows, Clayton, Hems, *J. Chem. Soc.*, 1949, *Suppl.* 185.

o-Tyrosol (o-β-*Hydroxyethylphenol*, o-*hydroxyphenylethyl alcohol*)



$C_8H_{10}O_2$ MW, 138

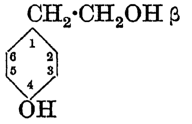
Oil. B.p. 168–9°/12 mm. Spar. misc. with cold H_2O. D^{18} 1·1531. n_D^{18} 1·5575. $FeCl_3$ ⟶ blue col.

2-*Et ether*: $C_{10}H_{14}O_2$. MW, 166. Oil. B.p. 136–7°.

Mono-acetyl deriv.: cryst. from ligroin. M.p. 64·5°. B.p. 170–80°/30 mm.

Stoermer, Kahlert, *Ber.*, 1901, **34**, 1809.

Tyrosol (p-*Hydroxyphenylethyl alcohol*, p-β-*hydroxyethylphenol*, p-*tyrosol*)



$C_8H_{10}O_2$ MW, 138

Needles from $CHCl_3$. M.p. 93°. B.p. about 310°, 195°/18 mm., 156°/15 mm. Very sol. H_2O, EtOH, Et_2O, Me_2CO, AcOH. Sol. $CHCl_3$, C_6H_6, CS_2. Spar. sol. pet. ether. $FeCl_3$ ⟶ blue col. Hot conc. H_2SO_4 ⟶ red col. H·CHO + H_2SO_4 ⟶ yellowish-green sol. ⟶ green ppt. with H_2O.

4-*Me ether*: $C_9H_{12}O_2$. MW, 152. Plates. M.p. 24°. B.p. 143°/13 mm. Insol. ligroin. *Acetyl*: b.p. 277–8°, 156–7°/11 mm. D^0 1·101.

4-*Et ether*: $C_{10}H_{14}O_2$. MW, 166. M.p. about 40°. B.p. 135–40°/7 mm.

β-*Acetyl*: prisms from Et_2O–ligroin. M.p. 59°. B.p. 192°/18 mm.

Diacetyl: oil. B.p. 187°/18 mm.

Dibenzoyl: needles from EtOH. M.p. 111°.

v. Braun, *Ber.*, 1912, **45**, 1283.

Neubauer, Fromherz, *Z. physiol. Chem.*, 1910, **70**, 342.

Siefken, *Ann.*, 1949, **562**, 75.

Tyrothricin.

The name given to the alcohol soluble fraction of the acid ppt. from a culture-medium of *Bacillus brevis*. Consists of a mixture of Gramicidin and Tyrocidine.

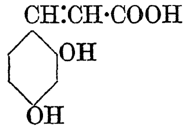
Hotchkiss, Dubos, *J. Biol. Chem.*, 1941, **141**, 155.

Goldstein, *Chem. Abstracts*, 1949, **43**, 3884.

U

Ulexine.
See Cytisine.

Umbellic Acid (2 : 4-*Dihydroxycinnamic acid*)



$C_9H_8O_4$ MW, 180

Yellow powder. Darkens at 240°, decomp. at 260°. Sol. EtOH, warm H_2O. Insol. Et_2O, C_6H_6, ligroin. Readily decomp. $k = 1{\cdot}88 \times 10^{-5}$ at 25°. Pb and Cu salts insol. H_2O.

Me ester : $C_{10}H_{10}O_4$. MW, 194. *Di-Me ether* : $C_{12}H_{14}O_4$. MW, 222. Needles from EtOH.Aq. M.p. 87°. Distils above 360°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. ligroin.

Et ester : $C_{11}H_{12}O_4$. MW, 208. *Di-Me ether* : $C_{13}H_{16}O_4$. MW, 236. Needles from pet. ether. M.p. 61°. B.p. 208–12°/13 mm. Sol. EtOH, Et_2O. Spar. sol. pet. ether.

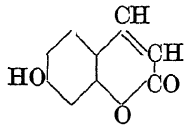
4-*Me ether* : $C_{10}H_{10}O_4$. MW, 194. Needles from H_2O. M.p. 180–5° decomp.

Di-Me ether : $C_{11}H_{12}O_4$. MW, 208. Two forms. (α-) Needles from EtOH. M.p. 138°. Sol. EtOH, Et_2O, C_6H_6. Heat. alone or with HCl ⟶ β-form. (β-). Needles from H_2O or EtOH.Aq. M.p. 186°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Insol. ligroin. Sol. H_2SO_4 ⟶ yellow col. Sublimes.

Di-Et ether : $C_{13}H_{16}O_4$. MW, 236. Two forms. (α-) Plates from EtOH.Aq. M.p. 106·5°. Sol. EtOH, Et_2O, C_6H_6. Spar. sol. H_2O. Heat ⟶ β-form with part. decomp. (β-) Cryst. from EtOH. M.p. 200°. Sol. Et_2O. Spar. sol. H_2O.

Will, Beck, *Ber.*, 1886, **19**, 1778.
Posen, *Ber.*, 1881, **14**, 2745.
Limaye, Kulkarni, *Chem. Abstracts*, 1942, **36**, 1033.

Umbelliferone (7-*Hydroxycoumarin*)



$C_9H_6O_3$ MW, 162

Occurs widely in plants. Needles from H_2O. M.p. 223–4°. Sol. 100 parts boiling H_2O. Very sol. EtOH, $CHCl_3$. Sol. AcOH, HCl. Spar. sol. Et_2O. Sol. alkalis and conc. H_2SO_4 with blue fluor. Sublimes. Forms Na salt. Reduces warm $AgNO_3$.

Me ether : *see* Herniarin.

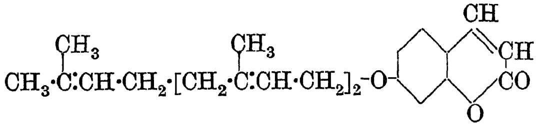
Et ether : $C_{11}H_{10}O_3$. MW, 190. Plates. M.p. 88°. Very sol. EtOH, AcOH, C_6H_6.

Acetyl : prisms or needles from H_2O. M.p. 140°.

d-*Glucoside* : *see* Skimmin.

Grimaux, *Bull. soc. chim.*, 1895, **13**, 900.
Gaind, Gupta, Ray, *Chem. Abstracts*, 1937, **31**, 7415.
Limaye, Munje, *Chem. Abstracts*, 1938, **32**, 2096.
Limaye, Joshi, *Chem. Abstracts*, 1942, **36**, 1010.
Nesmeyanov *et al.*, *J. Gen. Chem. U.S.S.R.*, 1937, **7**, 2767.

Umbelliprenin (*Umbelliferone farnesyl ether*)

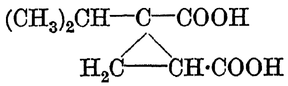


$C_{24}H_{30}O_3$ MW, 366

Occurs in angelica seed (*Angelica archangelica*). Cryst. from Et_2O–pet. ether. M.p. 61–3°. Cold AcOH–H_2SO_4 ⟶ umbelliferone.

Späth, Vierhapper, *Ber.*, 1938, **71**, 1667; *Monatsh.*, 1938, **72**, 179.

Umbellularic Acid (1-*Isopropylcyclopropane*-1 : 2-*dicarboxylic acid*)



$C_8H_{12}O_4$ MW, 172

Cis :

dl-.

Needles from C_6H_6–pet. ether. M.p. 126–7°. Also labile form, m.p. 117–18°.

p-*Phenylphenacyl ester* : needles from AcOH. M.p. 116–17°.

Anhydride : b.p. 140°/20 mm.

d-.

Needles + $1H_2O$ from H_2O, m.p. 78–9°. Prisms from C_6H_6–pet. ether, m.p. 119–20°. $[\alpha]_D^{16}$ + 86·9° in $CHCl_3$.

Acid brucine salt : plates from H_2O. M.p. 110–20°. $[\alpha]_{5461}^{20}$ + 6·2° in Me_2CO.

l-.

Needles + $1H_2O$ from H_2O, m.p. 78–9°. Prisms from C_6H_6–pet. ether, m.p. 119–20°. $[\alpha]_D^{13}$ − 88·8° in $CHCl_3$.

Acid brucine salt : needles from H_2O. $[\alpha]_{5461}^{16}$ − 29·5° in Me_2CO.

Trans :

dl-.

Prisms from H_2O. M.p. 197°. Sol. Me_2CO, AcOEt. Spar. sol. C_6H_6, $CHCl_3$. Heat alone or with CH_3COCl or Ac_2O ⟶ *cis*-anhydride.

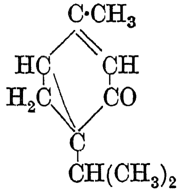
p-*Phenylphenacyl ester*: needles from AcOEt or Me_2CO. M.p. 175–6°.
Di-Et ester: b.p. 130–5°/20 mm.

d-.
Needles from H_2O. M.p. 155°. $[\alpha]^{21}_{5461}$ + 232·1° in Me_2CO.
Brucine salt: needles from H_2O. $[\alpha]^{22}_{5461}$ − 15·4° in $CHCl_3$.

l-.
Needles from H_2O. M.p. 153°. $[\alpha]^{21}_{5461}$ − 236·2° in Me_2CO.
Brucine salt: prisms from H_2O. M.p. 149–51° decomp. $[\alpha]^{25}_{5261}$ − 98·8° in $CHCl_3$.

Phillips, Ramage, Simonsen, *J. Chem. Soc.*, 1936, 828.
Rydon, *ibid.*, 829.
Guha, Muthanna, *Ber.*, 1938, **71**, 2668.

Umbellulone

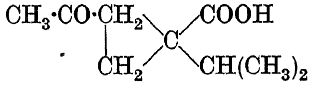


$C_{10}H_{14}O$ MW, 150

Constituent of essential oil from *Umbellularia californica*, Meissn. Colourless oil. B.p. 219–20°/749 mm., 92·5–93°/10 mm. D^{15}_{15} 0·9581. n^{20}_{D} 1·48325. $[\alpha]_D$ − 37°.
Semicarbazone: cryst. from MeOH. M.p. 238–9° decomp. (rapid heat. 240–2°).
Thiosemicarbazido-thiosemicarbazone: m.p. 210–11°.

Power, Lees, *J. Chem. Soc.*, 1904, **85**, 636.
Gillam, West, *J. Chem. Soc.*, 1945, 95.
Aries, Kidder, *Chem. Abstracts*, 1948, **42**, 9090.

Umbellulonic Acid (1-*Isopropyl*-2-*aceto-cyclopropane*-1-*carboxylic acid*)

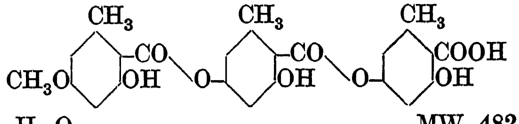


$C_9H_{14}O_3$ MW, 170

Prisms from pet. ether. M.p. 102°. $[\alpha]_D$ + 378° in $CHCl_3$. Sol. Et_2O, pet. ether. Spar. sol. H_2O.
Et ester: b.p. 238–9°.
Oxime: prisms from AcOEt. M.p. 169–70°.

Tutin, *J. Chem. Soc.*, 1906, **89**, 1104.

Umbilicaric Acid (*Monomethyl ether of gyrophoric acid*)

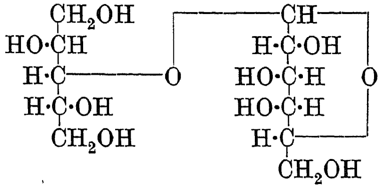


$C_{25}H_{22}O_{10}$ MW, 482

Occurs in numerous lichens. Plates from EtOH. M.p. 185–6°. Sol. EtOH, Et_2O, Me_2CO, $CHCl_3$. Spar. sol. Me_2CO.Aq. Insol. H_2O. Alc. $FeCl_3$ ⟶ violet col.
Triacetyl: m.p. 193–4° (197°). *Me ester*: prisms from Me_2CO.Aq. M.p. 206°.

Asahina, Yosioka, *Ber.*, 1937, **70**, 204.
Koller, Pfeiffer, *Monatsh.*, 1933, **62**, 359.

Umbilicin (3-β-D-*Galactopyranosido*-D-*arabitol*)



Suggested structure

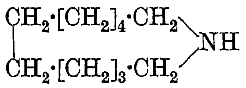
$C_{11}H_{22}O_{10}$ MW, 314

Occurs in the lichen *Umbilicaria pustulata*, (L.) Hoffm. Cryst. from EtOH. M.p. 138–9°. $[\alpha]^{20}_{D}$ − 81° (c = 2 in H_2O). Hyd. ⟶ D-arabitol + D-galactose.
Octa-acetyl: cryst. from MeOH. M.p. 84–5°. $[\alpha]^{20}_{D}$ − 20° (c = 2 in $CHCl_3$).

Lindberg, Wachtmeister, Wickberg, *Acta Chem. Scand.*, 1952, **6**, 1052.

1 : 7-Undecadiene-1-carboxylic isobutyl-amide.
See Herculin.

Undecamethyleneimine



$C_{11}H_{23}N$ MW, 169

M.p. 41–2°. pK 9·14.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1949, **32**, 544.

Undecanal.
See Undecyl Aldehyde.

Undecane (*Hendecane*)

$CH_3·[CH_2]_9·CH_3$

$C_{11}H_{24}$ MW, 156

Constituent of natural petroleum. F.p. − 25·6°. B.p. 194·5°, 81°/15 mm., 61°/2 mm. D^{20}_{4} 0·7411. $n^{19·5}_{D}$ 1·41862.

Hess, Bappert, *Ann.*, 1925, **441**, 151.
Clemmensen, *Ber.*, 1913, **46**, 1841.

Undecane-1-carboxylic Acid.
See Lauric Acid.

Undecane-1 : 11-dicarboxylic Acid.
See Brassylic Acid.

Undecanol-1.
See Undecyl Alcohol.

Undecanol-2.
See Methylnonylcarbinol.

Undecanol-3.
See Ethyloctylcarbinol.

Undecanol-5.
See Butyl-*n*-hexylcarbinol.
Undecanol-6.
See Di-*n*-amylcarbinol.
Undecanone-2.
See Methyl nonyl Ketone.
Undecanone-3.
See Ethyl octyl Ketone.
Undecanone-4.
See Propyl heptyl Ketone.
Undecanone-5.
See Butyl *n*-hexyl Ketone.
Undecanone-6.
See *n*-Caprone.
Undecene.
See Undecylene.

1-Undecenol-10

$CH_3 \cdot CH(OH) \cdot [CH_2]_7 \cdot CH{:}CH_2$

$C_{11}H_{22}O$ MW, 170

Constituent of essential oil of *Litsea odorifera*, Val. B.p. 233°. D^{10} 0·835.

Romburgh, *Chem. Zentr.*, 1911, II, 1863.

1-Undecenol-11.
See Undecenyl Alcohol.

2-Undecenol-11 (*Isoundecylenic alcohol*)

$HOCH_2 \cdot [CH_2]_7 \cdot CH{:}CH \cdot CH_3$

$C_{11}H_{22}O$ MW, 170

Liq. B.p. 248·5°, 124–5°/8 mm. D^{15} 0·8507. n_D^{19} 1·4535.
Acetyl: b.p. 127–8°/7 mm. D^{15} 0·8841.
Phenylurethane: needles from EtOH. M.p. 51–2°.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1926, **9**, 1085.

Undecenyl Alcohol (1-*Undecenol*-11, *undecylenic alcohol*)

$HO \cdot CH_2 \cdot [CH_2]_8 \cdot CH{:}CH_2$

$C_{11}H_{22}O$ MW, 170

Liq. solidifying to leaflets on cooling. F.p. – 2°. B.p. 250°, 132–3°/15 mm., 122°/3 mm. D^{15} 0·8495. n_D^{19} 1·4506.
Acetyl: liq. B.p. 125–7°/7 mm. D^{20} 0·8808. n_D^{20} 1·4390.
Phenylurethane: needles from EtOH. M.p. 54·5–55°.
Allophanate: m.p. 143°.

Grün, Wirth, *Ber.*, 1922, **55**, 2208.
Bouveault, Blanc, *Bull. soc. chim.*, 1904, **31**, 1210.

Undecenylamine (11-*Amino*-1-*undecylene*, 11-*amino*-1-*undecene*)

$H_2N \cdot CH_2 \cdot [CH_2]_8 \cdot CH{:}CH_2$

$C_{11}H_{23}N$ MW, 169

Liq. B.p. 238–40°, 123°/16 mm.
N-*Benzoyl*: plates from C_6H_6. M.p. 41–2°.

Krafft, Tritschler, *Ber.*, 1900, **33**, 3581.

Undecoic Acid.
See Undecylic Acid.

Undecyl Alcohol (*Undecanol*-1, *hendecyl alcohol, hendecanol*-1)

$CH_3 \cdot [CH_2]_9 \cdot CH_2OH$

$C_{11}H_{24}O$ MW, 172

F.p. 19° (11°). B.p. 147°/25 mm., 131°/15 mm., 123–5°/6 mm. D_4^{23} 0·8334. n_D^{23} 1·4392.
p-*Nitrobenzoyl*: m.p. 29·2°.
3 : 5-*Dinitrobenzoyl*: m.p. 55°.
3-*Nitrophthaloyl*: m.p. 123·2–123·3°.
Phenylurethane: needles from EtOH. M.p. 62°.
o-*Nitrophenylurethane*: m.p. 37°.
m-*Nitrophenylurethane*: plates from EtOH. M.p. 56°.
p-*Nitrophenylurethane*: m.p. 99·5°.
3 : 5-*Dinitrophenylurethane*: m.p. 62°.
Allophanate: cryst. from EtOH. M.p. 155·5–156°.

Ford, Marvel, *Organic Syntheses*, 1930, X, 63.
Levene, West, Allen, Scheer, *J. Biol. Chem.*, 1915, **23**, 72.
Hoeke, *Rec. trav. chim.*, 1935, **54**, 505.
Dickinson *et al.*, *J. Am. Chem. Soc.*, 1937, **59**, 1094.

Undecyl Aldehyde (*Undecylic aldehyde, undecanal*)

$CH_3 \cdot [CH_2]_9 \cdot CHO$

$C_{11}H_{22}O$ MW, 170

F.p. – 4°. B.p. 116–17°/18 mm. D_4^{23} 0·8251. n_D^{23} 1·4322. Readily polymerises. Oxidises in air to undecylic acid.
Oxime: needles from MeOH.Aq. M.p. 72°.
Semicarbazone: cryst. from MeOH. M.p. 103°.
Azine: cryst. from MeOH. M.p. 57°.
2 : 4-*Dinitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 104°.

Darzens, Levy, *Compt. rend.*, 1933, **196**, 348.
Blaise, Guerin, *Bull. soc. chim.*, 1903, **29**, 1202.
Blaise, *Bull. soc. chim.*, 1904, **31**, 492.
Allen, Gates, *J. Org. Chem.*, 1941, **6**, 596.
Davies, Hodgson, *J. Soc. Chem. Ind.*, 1943, **62**, 128.

***n*-Undecylamine** (1-*Aminoundecane, hendecylamine*)

$CH_3 \cdot [CH_2]_9 \cdot CH_2NH_2$

$C_{11}H_{25}N$ MW, 171

M.p. 15–16°. B.p. 231–2°/727 mm., 129·5–130·5°/25 mm. Sol. hot H_2O, EtOH. Insol. Et_2O. Volatile in steam.
B,HCl: cryst. from conc. HCl. M.p. 190°.
B_2,H_2PtCl_6: yellow plates from H_2O. Decomp. about 180°.
N-*Acetyl*: needles from pet. ether. M.p. 48°. B.p. 192°/12 mm. *B,HCl*: cryst. M.p. 65–6°.
N-*Benzoyl*: needles from C_6H_6–ligroin. M.p. 60°.

Naegeli, Grüntuch, Lendorff, *Helv. Chim. Acta*, 1929, **12**, 227.
Jeffreys, *Am. Chem. J.*, 1899, **22**, 33.

sec.-n-Undecylamine (*2-Aminoundecane*)

$CH_3\cdot[CH_2]_8\cdot CH(NH_2)\cdot CH_3$

$C_{11}H_{25}N$ MW, 171

B.p. 231°/741 mm., 113°/26 mm. Absorbs CO_2 readily.

B,HCl: needles from ligroin. M.p. 84°.

B_2,H_2PtCl_6: yellow tablets from EtOH. Darkens at about 240°.

N-*Acetyl*: needles from EtOH.Aq. M.p. 58°.

Picrate: m.p. 111°.

Thoms, Mannich, *Ber.*, 1903, **36**, 2554.

1-Undecylene (*Undecene*-1, *hendecene*-1)

$CH_3\cdot[CH_2]_8\cdot CH{:}CH_2$

$C_{11}H_{22}$ MW, 154

B.p. 192–5°, 84°/18 mm. D_{20}^{20} 0·7787. n_D 1·4440.

Ruhemann, *Z. angew. Chem.*, 1931, **44**, 78.
Treibs, *Chem. Abstracts*, 1940, **34**, 7843.

2-Undecylene (*Undecene*-2, *hendecene*-2)

$CH_3\cdot[CH_2]_7\cdot CH{:}CH\cdot CH_3$

$C_{11}H_{22}$ MW, 154

B.p. 192–3°, 78·5°/14 mm. D_{15}^{15} 0·7735. n_D 1·43325.

Thoms, Mannich, *Ber.*, 1903, **36**, 2548.
Moretti, *Compt. rend.*, 1948, **226**, 343.

4-Undecylene-1-carboxylic Acid.
See Linderic Acid.

1-Undecylenic Acid

$CH_3\cdot[CH_2]_7\cdot CH{:}CH\cdot COOH$

$C_{11}H_{20}O_2$ MW, 184

Formed on dehydration of castor oil.

Nitrile: $C_{11}H_{19}N$. MW, 165. Two stereoisomeric forms. (i) B.p. 119·7–119·9°/10 mm. D_4^{20} 0·83255. n_D^{20} 1·44816. (ii) B.p. 127·9–128·1°/10 mm. D_4^{20} 0·83359. n_D^{20} 1·45146.

Amide: $C_{11}H_{21}ON$. MW, 183. Two stereoisomeric forms corresponding to the above nitriles. (i) Cryst. from C_6H_6. M.p. 76–7°. (ii) Cryst. from C_6H_6. M.p. 114–15°.

Hydrazide: m.p. 84–5°. *B,HCl*: m.p. 110–12°.

Phenylhydrazide: m.p. 97°.

Caillie, *Chem. Zentr.*, 1936, I, 4423.
Roger, Dvolaitskaya, *Chem. Abstracts*, 1938, **32**, 1241.
Stempel, Schaffel, *J. Am. Chem. Soc.*, 1942, **64**, 470.

8-Undecylenic Acid

$CH_3\cdot CH{:}CH\cdot[CH_2]_7\cdot COOH$

$C_{11}H_{20}O_2$ MW, 184

Cis:

M.p. 1–2°. B.p. 118–20°/0·8 mm. n_D^{20} 1·4530.

Et ester: b.p. 88–90°/0·6 mm. n_D^{20} 1·4418.

p-*Phenylphenacyl ester*: plates from EtOH. M.p. 64·5–65°.

Trans:

M.p. 17–18°. B.p. 121–3°/0·7 mm. n_D^{20} 1·4519.

Et ester: b.p. 87–9°/0·5 mm. n_D^{20} 1·4406.

Ames, Bowman, *J. Chem. Soc.*, 1952, 677.

9-Undecylenic Acid

$CH_2{:}CH\cdot[CH_2]_8\cdot COOH$

$C_{11}H_{20}O_2$ MW, 184

Cryst. M.p. 24·5°. B.p. 275°, 165°/15 mm. D_{25}^{25} 0·9102. n_α^{24} 1·44642.

Cu salt: m.p. 232–4°.

Pb salt: m.p. 80°.

Zn salt: m.p. 115–16°.

Me ester: $C_{12}H_{22}O_2$. MW, 198. M.p. — 27·5°. B.p. 248°, 124°/10 mm. D^{15} 0·889. n_D^{20} 1·43928.

Et ester: $C_{13}H_{24}O_2$. MW, 212. B.p. 263·5–265·5°, 131·5°/16 mm. D_{25}^{25} 0·87658. n_D^{23} 1·4449.

Anhydride: $C_{22}H_{38}O_3$. MW, 350. M.p. 13–13·5°.

Chloride: $C_{11}H_{19}OCl$. MW, 202·5. Liq. B.p. 128·5°/14 mm.

Amide: $C_{11}H_{21}ON$. MW, 183. Plates from EtOH. M.p. 87°.

Nitrile: $C_{11}H_{19}N$. MW, 165. Liq. B.p. 257°, 129–30°/14 mm.

Krafft, Brunner, *Ber.*, 1884, **17**, 2985.

Undecylenic Alcohol.
See Undecenyl Alcohol.

Undecylic Acid (*Undecoic acid, hendecanoic acid*)

$CH_3\cdot[CH_2]_9\cdot COOH$

$C_{11}H_{22}O_2$ MW, 186

Cryst. from Me_2CO. M.p. 28·2–28·6°. B.p. 212·5°/100 mm., 164°/15 mm. Ag salt insol. H_2O. Ba salt spar. sol. H_2O.

Me ester: $C_{12}H_{24}O_2$. MW, 200. B.p. 123°/9–10 mm.

Et ester: $C_{13}H_{26}O_2$. MW, 214. B.p. 140°/20 mm.

p-*Chlorophenacyl ester*: plates from EtOH. M.p. 60·2°.

p-*Bromophenacyl ester*: plates from EtOH. M.p. 68·2°.

p-*Iodophenacyl ester*: plates from EtOH. M.p. 81·8°.

p-*Phenylphenacyl ester*: cryst. from EtOH. M.p. 79·5–80°.

Anhydride: cryst. from Me_2CO. M.p. 35°. B.p. 210–45°/15–20 mm.

Amide: $C_{11}H_{23}ON$. MW, 185. Cryst. from EtOH. M.p. 98–98·7°.

Nitrile: $C_{11}H_{21}N$. MW, 167. Liq. B.p. 253–4°.

Anilide: cryst. from EtOH. M.p. 71°.

o-*Toluidide*: m.p. 78°.

p-*Toluidide*: cryst. from EtOH. M.p. 80°.

Hydrazide: m.p. 101–2°.

Phenylhydrazide: m.p. 110°.

Moses, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 2101.
Asano, *Chem. Abstracts*, 1922, **16**, 1931.
Robertson, *J. Chem. Soc.*, 1919, **115**, 1210.
Levene, West, *J. Biol. Chem.*, 1914, **18**, 464.
Price, Griffith, *J. Am. Chem. Soc.*, 1940, **62**, 2884.
Rodier, *Bull. soc. chim.*, 1948, 637.
Cason *et al.*, *J. Org. Chem.*, 1950, **15**, 139.

Undecyl phenyl Ketone.
See Laurophenone.

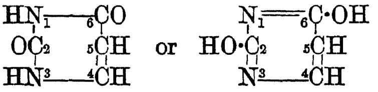
1-Undecyne.
See Nonylacetylene.

2-Undecyne.
See Methyloctylacetylene.

5-Undecyne.
See Butylamylacetylene.

Ungernine.
See Tazettine.

Uracil (2 : 6-*Dihydroxypyrimidine*, 2 : 6-*pyrimidinedione*)



$C_4H_4O_2N_2$ MW, 112

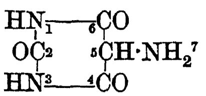
Needles from H_2O. M.p. 335°. Sol. hot H_2O, ammonia. Spar. sol. cold H_2O. Forms K salt.
N-*Me*: *see* Methyluracil.
N-*Di-Me*: *see* Dimethyluracil.
1-N-*Et*: $C_6H_8O_2N_2$. MW, 140. Prisms from C_6H_6. M.p. 173–4° (147·5°). Sol. hot H_2O, EtOH.
1-N-*Et*-3-N-*Me*: *see* Methylethyluracil.
1 : 3-N-*Di-Et*: $C_8H_{12}O_2N_2$. MW, 168. M.p. 14–15°. B.p. 290–5°, 135°/4 mm.
1-N-*Benzyl*: $C_{11}H_{10}O_2N_2$. MW, 202. Prisms from H_2O. M.p. 175°.
3-N-*Benzyl*: prisms from EtOH. M.p. 173°. Very sol. hot AcOH. Sol. Me_2CO. Spar. sol. H_2O, $CHCl_3$. Insol. Et_2O, cold C_6H_6, dil. min. acids.

Hilbert, Johnson, *J. Am. Chem. Soc.*, 1930, **52**, 2001.
Davidson, Baudisch, *J. Am. Chem. Soc.*, 1926, **48**, 2382.
Gabriel, *Ber.*, 1905, **38**, 1690.
Hilbert, *J. Am. Chem. Soc.*, 1937, **59**, 330.

Uracil-4-carboxylic Acid.
See Orotic Acid.

Uradal.
See Adalin.

Uramil (5-*Aminobarbituric acid*)

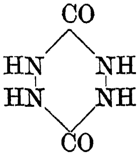


$C_4H_5O_3N_3$ MW, 143

Needles or plates from H_2O. Does not melt below 400°. Mod. sol. hot H_2O. Insol. cold H_2O, Et_2O, $CHCl_3$, CS_2, C_6H_6. Sol. conc. H_2SO_4, dil. aq. KOH, NH_3. Darkens in air. Forms Na, K, Ba, Pb salts. Pb salt spar. sol. H_2O.
1 : 3-N-*Di-Me*: $C_6H_9O_3N_3$. MW, 171. Needles. M.p. about 200° decomp. Turns red in air. Spar. sol. cold H_2O. Insol. EtOH. Sol. dil. acids. Reduces $AgNO_3$ and Fehling's. Decomp. by alkalis.
1 : 3 : 7-N-*Tri-Me*: $C_7H_{11}O_3N_3$. MW, 185. Needles from H_2O or EtOH. M.p. about 200° decomp. Sol. hot H_2O. Spar. sol. EtOH. Reduces $AgNO_3$.
1 : 3 : 7 : 7-N-*Tetra-Me*: $C_8H_{13}O_3N_3$. MW, 199. Cryst. from EtOH. M.p. 230–2° decomp. Mod. sol. H_2O. Spar. sol. MeOH, EtOH. Very spar. sol. Et_2O, C_6H_6. *Monohydrate*: needles from H_2O. M.p. 225° decomp.
7-N-*Et*: $C_6H_9O_3N_3$. MW, 171. Needles from H_2O. M.p. 297° decomp. Sol. hot H_2O.
1 : 3-N-*Di-Et*: $C_8H_{13}O_3N_3$. MW, 199. Cryst. M.p. about 200° decomp. Spar. sol. H_2O, EtOH, Et_2O, Me_2CO, C_6H_6. Sols decompose on heating.
1 : 3-N-*Diphenyl*: $C_{16}H_{13}O_3N_3$. MW, 295. Needles from EtOH. M.p. 97°. Decomp. in air, becoming red.
7-N-*Benzyl*: $C_{11}H_{11}O_3N_3$. MW, 233. Needles from AcOH. Decomp. about 280°.

Hartmann, Sheppard, *Organic Syntheses*, 1932, XII, 84.

***p*-Urazine** (*Dicarbamide, diurea*)

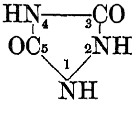


$C_2H_4O_2N_4$ MW, 116

Prisms from H_2O. M.p. about 270° (266°). Spar. sol. EtOH, AcOH, hot H_2O. Reduces $NH_3.AgNO_3$. $FeCl_3 \longrightarrow$ red col. $H_2SO_4 + HNO_3 \longrightarrow$ violet-red col. Acts as monobasic acid.
Hydrazine salt: prisms. M.p. 197°.
N-*Acetyl*: plates. Decomp. at 235°.

Purgotti, Viganò, *Gazz. chim. ital.*, 1901, **31**, ii, 550.
Linch, *J. Chem. Soc.*, 1912, **101**, 1756.

Urazole (3 : 5-*Diketodihydro*-1 : 2 : 4-*triazole*, *hydrazodicarbonimide*, 3 : 5-*diketopyrazolidine*)



$C_2H_3O_2N_3$ MW, 101

Plates from H_2O. M.p. 244° decomp. Very sol. H_2O. Spar. sol. EtOH. Insol. Et_2O. Reacts acid.

Acetyl deriv.: cryst. from H_2O. M.p. 221·5°.

Diacetyl deriv.: plates from EtOH. M.p. 206°.

Triacetyl deriv.: prisms from C_6H_6. M.p. 138°.

1-*Me*: m.p. 241°.

1-*Me*-2-*phenyl*: m.p. 183°.

4-*Me*-1-*phenyl*: m.p. 223°.

4-*Phenyl*: m.p. 202–3°.

1 : 4-*Diphenyl*: cryst. from EtOH. M.p. 160°.

Stollé, Krauch, *J. prakt. Chem.*, 1913, **88**, 314.
Thiele, Stange, *Ann.*, 1894, **283**, 41.
Arndt *et al.*, *Chem. Abstracts*, 1948, **42**, 8190.
Loewe, Türgen, *Chem. Abstracts*, 1950, **44**, 6415.

Urea (*Carbamide, isourea*)

$NH_2 \cdot CO \cdot NH_2$ or $NH_2 \cdot C(OH){:}NH$
Iso-.

CH_4ON_2 MW, 60

Constituent of blood and tissue fluids of all vertebrates and of the urine of all mammals. Also occurs in many invertebrates, Nematodes, Crustaceans, Molluscs, etc., and in many fungi and moulds. Prisms with faint salty taste from H_2O or EtOH. M.p. 132°. Sol. MeOH, EtOH. Insol. Et_2O, $CHCl_3$, C_6H_6. Sol. 1 part H_2O at 17° ⟶ neutral sol. Sublimes without decomp. in cathode-ray vacuum. Heat above m.p. ⟶ cyanuric acid: at 150–70° ⟶ biuret: at 200° ⟶ triureide of cyanuric acid. Cold aq. alkalis ⟶ NH_3. Hot dil. acids or alkalis ⟶ $CO_2 + NH_3$. *p*-Dimethylaminobenzaldehyde + HCl (Ehrlich's reagent) ⟶ intense yellow col. After heating ⟶ biuret reaction with NaOH and $CuSO_4$. HNO_2 or alk. NaOBr ⟶ evolution of N.

B,HCl: plates. Decomp. at 145°.

B,HNO₃: prisms. M.p. 152° decomp.

N : N′-*Diacetyl*: needles from EtOH. M.p. 152–3°.

N : N′-*Dibenzoyl*: needles from EtOH. M.p. 218–22° decomp.

N-*Dibenzoyl*: needles from EtOH. M.p. 197°.

Benzenesulphonyl: m.p. 169°.

p-*Toluenesulphonyl*: m.p. 192°.

N-*Tri-Me*: $C_4H_{10}ON_2$. MW, 102. Prisms from Et_2O. M.p. 75·5°. B.p. 232·5°/764·5 mm. Very sol. H_2O, EtOH.

N-*Tetra-Me*: $C_5H_{12}ON_2$. MW, 116. B.p. 177·5°/766 mm. Very sol. EtOH, Et_2O.

N-*Tri-Et*: $C_7H_{16}ON_2$. MW, 144. Prisms from pet. ether. M.p. 65°. B.p. 223° (235°).

N-*Tetra-Et*: $C_9H_{20}ON_2$. MW, 172. Liq. with peppermint odour. B.p. 210–15° (205°). Sol. acids, re-ppd. by alkalis. Insol. H_2O.

N-*Triphenyl*: $C_{19}H_{16}ON_2$. MW, 288. Plates. M.p. 136°.

N-*Tetraphenyl*: $C_{25}H_{20}ON_2$. MW, 364. Cryst. from C_6H_6. M.p. 183°. Sol. EtOH.

O-*Me*: $C_2H_6ON_2$. MW, 74. M.p. 44–5°. B.p. 82°/9 mm. $k = 6{\cdot}4 \times 10^{-5}$ at 25°. Volatile in vapours of EtOH, Et_2O. *B,HCl*: m.p. 130° decomp. B_2,H_2PtCl_6: m.p. 178° decomp. *Picrate*: m.p. 184° decomp. N-*Acetyl*: cryst. from pet. ether. M.p. 58·5°. N-*Benzoyl*: prisms from MeOH.Aq. M.p. 77–8°.

O-*Et*: $C_3H_8ON_2$. MW, 88. M.p. 42°. B.p. 95–6°/15 mm. Sol. Et_2O. $k = 10{\cdot}4 \times 10^{-5}$ at 25°. *B,HCl*: m.p. 123–4° decomp. B_2,H_2PtCl_6: yellow plates from EtOH. M.p. 178·5° decomp. N-*Benzoyl*: cryst. from EtOH.Aq. M.p. 74–5°.

Other urea derivatives are given separately elsewhere.

Foss, *L'Urée* (Paris, 1928).
Werner, *The Chemistry of Urea*, (Monographs on Biochemistry, London, 1923).
Michler, Escherich, *Ber.*, 1879, **12**, 1164.
Wallach, *Ann.*, 1882, **214**, 275.
Schenck, *Z. physiol. Chem.*, 1912, **77**, 383.
Reudler, *Rec. trav. chim.*, 1914, **33**, 64.
Stoughton *et al.*, *J. Am. Chem. Soc.*, 1939, **61**, 408.
Pearl, Dehn, *J. Am. Chem. Soc.*, *ibid.*, 1377.
Cox, *J. Am. Chem. Soc.*, 1940, **62**, 743.
Bellavita, Cagnoli, *Gazz. chim. ital.*, 1939, **69**, 583.

Urea chloride.
See under Carbamic Acid.

Ureidoacetic Acid.
See Hydantoic Acid.

5-Ureidobarbituric Acid.
See ψ-Uric Acid.

Ureidoethyl Alcohol.
See *N*-2-Hydroxyethylurea.

Ureidoformamide.
See Biuret.

Ureidoformic Acid.
See Allophanic Acid.

Ureidonorleucine.
See Homocitrulline.

***p*-Ureidophenetole.**
See Dulcin.

Ureidophenylacetic Acid.
See 1-Phenylhydantoic Acid.

1-Ureidopropionic Acid (N-*Carbamyl-α-alanine*, N-*carbamyl*-1-*aminopropionic acid*)

$$NH_2 \cdot CO \cdot NH \cdot \underset{}{\overset{CH_3}{\overset{|}{C}}}H \cdot COOH$$

$C_4H_8O_3N_2$ MW, 132

l-.

Prisms from H_2O. M.p. 198–200°. Sol. 70 parts cold H_2O. $[\alpha]_D^{20} - 9{\cdot}6°$ in H_2O. Hot dil. HCl ⟶ *l*-5-methylhydantoin.

dl-.
Prisms, needles or plates from H_2O, plates from EtOH. M.p. 185° (161° decomp.). Sol. 100 parts EtOH at 20° and 46 parts H_2O at 20°. Insol. Et_2O. Heat at 140° or with dil. HCl ⟶ 5-methylhydantoin.
K salt: needles + $1H_2O$. Decomp. at 200–5°.

Dakin, *J. Chem. Soc.*, 1915, **107**, 439.

2-Ureidopropionic Acid (N-*Carbamyl*-2-*aminopropionic acid*)

$NH_2 \cdot CO \cdot NH \cdot CH_2 \cdot CH_2 \cdot COOH$

$C_4H_8O_3N_2$ MW, 132

Cryst. M.p. 170–1° decomp. Sol. H_2O. Spar. sol. most org. solvents. Heat at 160–70° or with dil. HCl ⟶ 2 : 4-diketohexahydropyrimidine.
K salt: cryst. Sinters at 80°, m.p. 100°.

Lengfeld, Stieglitz, *Am. Chem. J.*, 1893, **15**, 516.

Urethane (*Ethyl carbamate, aminoformic acid ethyl ester*)

$NH_2 \cdot CO \cdot OC_2H_5$

$C_3H_7O_2N$ MW, 89

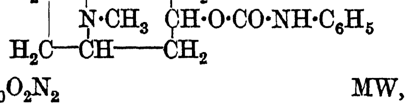
Prisms from C_6H_6 or toluene. M.p. 49–50°. B.p. 184°. Very sol. H_2O, EtOH, Et_2O, $CHCl_3$, C_6H_6. Spar. sol. ligroin. Heat aq. sol. to 130° ⟶ urea. Heat with NaOH ⟶ NaCNO + Na_2CO_3 + C_2H_5OH.
N-*Nitroso*: $C_3H_6O_3N_2$. MW, 118. Yellow needles from ligroin. M.p. 51–2° decomp. Very sol. MeOH, EtOH, Et_2O, Me_2CO. Sol. H_2O. Spar. sol. ligroin.
N-*Chloroacetyl*: cryst. from EtOH. M.p. 129°.
N-*Bromoacetyl*: cryst. from EtOH. M.p. 119°.
N-*Dichloroacetyl*: needles from H_2O. M.p. 98°.
N-p-*Nitrobenzoyl*: cryst. from C_6H_6. M.p. 152°.

Basterfield, Greig, *Chem. Zentr.*, 1933, II, 1021.
Basterfield, Woods, Wright, *J. Am. Chem. Soc.*, 1926, **48**, 2371.
Guerci, *Chem. Abstracts*, 1922, **16**, 2481.

Urethylan.
Methyl carbamate. *See under* Carbamic Acid.

Uretidone.
See Methyleneurea.

Uretropine (*Phenylcarbamic tropine ester, anilinoformyltropein*)

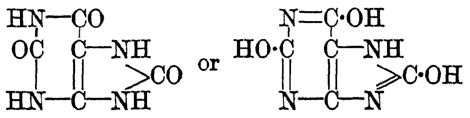


$C_{15}H_{20}O_2N_2$ MW, 260.

Prisms from Et_2O. M.p. 171–2°. Very sol. EtOH, Et_2O. Spar. sol. cold C_6H_6. Has mydriatic action.
B,HCl: prisms from EtOH.Aq. M.p. 289–90°.
B_2,H_2SO_4: needles + $4H_2O$ from H_2O. M.p. 201° decomp.
B,HAuCl₄: orange-red cryst. powder. M.p. 188–9°.
Picrate: yellow prisms from EtOH.Aq. M.p. 223–4°.

Jowett, Pyman, *J. Chem. Soc.*, 1909, **95**, 1027.

Uric Acid (2 : 6 : 8-*Trihydroxypurine*)

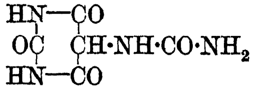


$C_5H_4O_3N_4$ MW, 168

Chief end-product of purine metabolism in man. Constituent of urine of animals, excrement of birds, reptiles, insects, etc. Rhombic prisms or plates. Insol. EtOH, Et_2O. Very spar. sol. H_2O. Sol. glycerol. Spar. sol. min. acids. Readily sol. alkalis. Heat ⟶ urea, cyanuric acid, HCN and NH_3. KOH fusion ⟶ KCN, KCNO, K_2CO_3 and $(COOK)_2$. HNO_3 ⟶ alloxan + urea. $KMnO_4$ ⟶ allantoin. Acts as weak dibasic acid. In alk. sol. reduces Ag and Cu salts, phosphomolybdates and phosphotungstates. Gives murexide reaction.

Traube, *Ber.*, 1900, **33**, 1371, 3035.
Fischer, *Ber.*, 1897, **30**, 559.

ψ-Uric Acid (5-*Ureidobarbituric acid*)

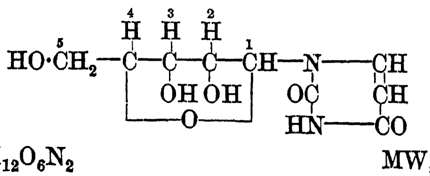


$C_5H_6O_4N_4$ MW, 186

Prisms. Spar. sol. H_2O. Sol. aq. alkalis. Heat of comb. C_v 455·2 Cal. HNO_3 ⟶ alloxan + urea. Alk. $KMnO_4$ ⟶ alloxanic acid. Heat with oxalic acid or with hot dil. HCl ⟶ uric acid.

Biltz, *Ann.*, 1921, **423**, 119.
Fischer, *Ber.*, 1897, **30**, 570, 3091.

Uridine



$C_9H_{12}O_6N_2$ MW, 244

Needles from EtOH.Aq. M.p. 165°. $[\alpha]_D^{20}$ + 4·0° in H_2O.
3-*Phosphate*: uridylic acid. *Dibrucine salt*: m.p. 177–81°. $[\alpha]_D$ − 60° in Py.
5-*Phosphate*: prisms from MeOH. M.p. 198·5°. $[\alpha]_D^{20}$ + 9·5° in H_2O.
N-*Me*: $C_{10}H_{14}O_6N_2$. MW, 258. Plates. M.p. 108–10°. Sol. MeOH, EtOH, Me_2CO, AcOH, H_2O, Py. Insol. Et_2O, C_6H_6.

2 : 3-*Di-Me ether*: $C_{11}H_{16}O_6N_2$. MW, 272. Cryst. from AcOEt. M.p. 168–9°. Sol. H_2O, EtOH, MeOH, AcOH, Me_2CO, Py. $[\alpha]_D^{24}$ + 68·0° in Me_2CO.

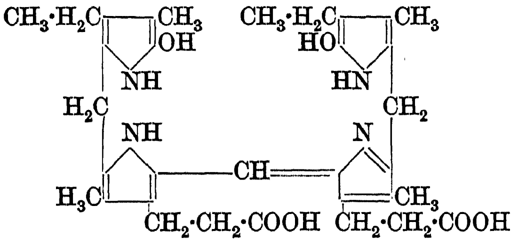
2 : 3-*Di*-p-*toluenesulphonyl*: needles from EtOH. M.p. 199°. $[\alpha]_D^{23}$ − 26·6° in Me_2CO.

2 : 3-*Benzylidene*: needles from H_2O. M.p. 189–90°.

Levene, Tipson, *J. Biol. Chem.*, 1934, **104**, 385; **105**, 419.
Levene, Jacobs, *Ber.*, 1910, **43**, 3158.
Gulland, Hobday, *J. Chem. Soc.*, 1940, 746.
Bredereck, Berger, *Ber.*, 1940, **73**, 1124.
Gulland, Smith, *J. Chem. Soc.*, 1947, 338.
Michelson, Todd, *J. Chem. Soc.*, 1949, 2476.
Brown, Haynes, Todd, *J. Chem. Soc.*, 1950, 3299.

Uridylic Acid.
See under Uridine.

Urobilin (*Stercobilin*)



$C_{33}H_{42}O_6N_4$ MW, 590

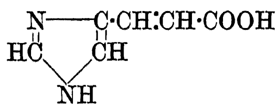
Also known as Urobilin IX-α. Constituent of urine and fæces. Yellow needles from $CHCl_3$ or MeCO. M.p. 177°. Very sol. MeOH, EtOH, AcOH, Py. Sol. Me_2CO, $CHCl_3$. Spar. sol. H_2O, Et_2O, AcOEt, C_6H_6. Insol. CCl_4, pet. ether. Very sol. amyl alcohol with green fluor. Very sol. conc. HCl and conc. H_2SO_4 ⟶ red sols.

B,HCl: prisms from Me_2CO, m.p. 199–200°: prisms from $CHCl_3$, m.p. 147–71°.

B,HBr: prisms from Me_2CO. M.p. 200–1°.

Siedel, Meier, *Z. physiol. Chem.*, 1936, **242**, 101.
Heilmeyer, Krebs, *Z. physiol. Chem.*, 1934, **228**, 33, 46.

Urocanic Acid (*Iminazolylacrylic acid*)



$C_6H_6O_2N_2$ MW, 138

Cis:
M.p. 175–6°.
B,HNO₃: m.p. 156°.
Picrolonate: m.p. 243°.

Trans:
Dihydrate. Cryst. from H_2O. M.p. 218–24°.
B,HNO₃: m.p. 190°.
Picrolonate: m.p. 268°.

Edlbacher, von Bidder, *Z. physiol. Chem.*, 1942, **276**, 126.
Edlbacher, Heitz, *Z. physiol. Chem.*, 1943, **279**, 63.

Uroporphyrin (*Urinoporphyrin*)

$C_{40}H_{38}O_{16}N_4(C_{41}H_{42}O_{16}N_4)$ MW, 830 (846)

Isolated from urine in a case of hæmatoporphyria congenita. Dark red fluor. cryst. + ½ Py from Py–EtOH.

Me ester: short needles from MeOH or $CHCl_3$. M.p. 295°. Forms Cu and Fe·· salts.

Et ester: long needles from MeOH or $CHCl_3$. M.p. 220°.

Fischer, *Z. physiol. Chem.*, 1915, **95**, 34.

Uropterin.
See Xanthopterin.

Uroselectan B.
See under 3 : 5-Di-iodo-1-methylchelidamic Acid.

Urotropine.
See Hexamethylenetetramine.

Uroxanic Acid (*Diureidomalonic acid*)

$$\begin{matrix}H_2N{\cdot}CO{\cdot}NH \\ H_2N{\cdot}CO{\cdot}NH\end{matrix}\!>C<\!\begin{matrix}COOH \\ COOH\end{matrix}$$

$C_5H_8O_6N_4$ MW, 220

Needles from C_6H_6. Decomp. at 162°. Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6. Insol. pet. ether. Loses CO_2 in H_2O at 60°. Ag salt spar. sol. H_2O.

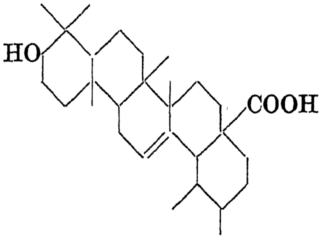
NH₄ salt: prisms. Decomp. at 182°.

Di-Et ester: $C_9H_{16}O_6N_4$. MW, 276. Cryst. from EtOH. M.p. 170°.

Biltz, Robl, *Ber.*, 1920, **53**, 1950.

Ursol P.
See p-Aminophenol.

Ursolic Acid (*Ursone, prunol, malol, malolic acid*)



Suggested structure

$C_{30}H_{48}O_3$ **MW, 456**

Constituent of leaves and skins of apples and pears, leaves of *Rhododendron hymenanthes*, leaves of *Epigaea asiatica*, Maxim, etc. Prisms from Et_2O. M.p. 285° (291°). Sol. hot AcOH, EtOH. Mod. sol. Et_2O, AcOH, Me_2CO, AcOEt, $CHCl_3$. Insol. H_2O, pet. ether. Liebermann-Salkowski reagent ⟶ blue col.

Me ester: $C_{31}H_{50}O_3$. MW, 470. Two forms. (i) Cryst. from EtOH.Aq. M.p. 170·5–171·5° (173°). Sol. cold Me_2CO. (ii) M.p. 230°. Insol. cold Me_2CO. *Benzoyl*: m.p. 215–16°. *Acetyl*: m.p. 246–7°.

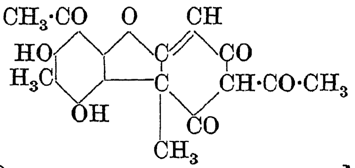
Phenacyl ester: m.p. 199–200°.

Drake, Duvall, *J. Am. Chem. Soc.*, 1936, **58**, 1687.
Fujii, Shimada, *J. Pharm. Soc. Japan*, 1935, **55**, 106.
Kuwada, Matsumoto, *Chem. Abstracts*, 1933, **27**, 3925.
Sando, *J. Biol. Chem.*, 1931, **90**, 477; 1923, **56**, 457.
Haar, *Rec. trav. chim.*, 1924, **43**, 542.
Kuwada, Matsukawa, *Chem. Abstracts*, 1934, **28**, 4739.
Ewen, Spring, *J. Chem. Soc.*, 1943, 523.
Jeger, *Fortschritte der Chemie organischer Naturstoffe*, Vol. 7, p. 59.

Ursone.

See Ursolic acid.

Usnic Acid



$C_{18}H_{16}O_7$ MW, 344

d-.

Occurs in various species of lichen. Constituent of Chinese drug "Shi-koa." Cryst. from MeOH or C_6H_6. M.p. 202–4° (195–6°). Very sol. $CHCl_3$. Sol. EtOH, Et_2O. Insol. H_2O. $[\alpha]_D^{20}$ + 468·8°.

Diacetyl: needles from MeOH. M.p. 199–200°. $[\alpha]_D^{16}$ + 205°. $FeCl_3 \longrightarrow$ red col.

l-.

M.p. 195–7°. Very sol. $CHCl_3$. Sol. EtOH, Et_2O. Insol. H_2O. $[\alpha]_D^{20}$ − 480°.

dl-.

M.p. 193–4°. $FeCl_3 \longrightarrow$ brown col.

Acetyl: m.p. 191°.

Diacetyl: m.p. 199–200°.

Curd, Robertson, *J. Chem. Soc.*, 1937, 894.
Asahina, Yanagita, *Ber.*, 1937, **70**, 66; 1936, **69**, 1646.
Shöpf, Kraus, Heuck, *Ann.*, 1927, **459**, 263.
Nakao, *Chem. Abstracts*, 1923, **17**, 3184.
Foster, Robertson, Healy, *J. Chem. Soc.*, 1939, 1594.
Schöpf, Ross, *Ann.*, 1941, **546**, 1.

Uteroverdin.

See Biliverdin.

Uvaleral.

See 1-Bromoisovalerylurea.

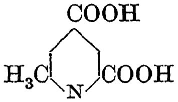
Uvinic Acid.

See Pyrotritaric Acid.

Uvitic Acid.

See 5-Methylisophthalic Acid.

Uvitonic Acid (*α-Picoline*-4 : 6-*dicarboxylic acid*, 6-*methyl-lutidinic acid*)



$C_8H_7O_4N$ MW, 181

Cryst. from H_2O. M.p. 282° decomp. Sol. hot AcOH, phenol, aniline. Sol. conc. H_2SO_4, HCl, NH_4OH. Spar. sol. hot $CHCl_3$, amyl alcohol. Very spar. sol. cold H_2O. Insol. CS_2, C_6H_6.

Böttinger, *Ber.*, 1880, **13**, 2032.

V

Vaccenic Acid.

The name assigned to an acid present in very small proportions in ox and sheep body fats and in butter fat. M.p. 39°. D_4^{70} 0·85601. n_C^{70} 1·43834; n_D^{70} 1·44071; n_F^{70} 1·44674. Believed to be a mixture of *trans*-9-octadecylenic acid and *trans*-10-octadecylenic acid.

Bertram, *Biochem. Z.*, 1928, **197**, 433.
Gupta, Hilditch, Paul, Shrivastava, *J. Chem. Soc.*, 1950, 3484.

Vaccinin (6-*Benzoyl*-d-*glucose*)

$\overline{CH(OH)\cdot[CH(OH)]_3\cdot CH}\cdot CH_2O\cdot CO\cdot C_6H_5$ (ring closed through O)

$C_{13}H_{16}O_7$ MW, 284

Amorph. hygroscopic mass with bitter taste. Cryst. from Me_2CO as monohydrate. M.p. 104–6°. Sol. H_2O, MeOH, EtOH, AcOEt, Me_2CO, Py. Spar. sol. $CHCl_3$, C_6H_6. Insol. Et_2O, pet. ether. $[\alpha]_D^{21}$ + 48° in EtOH, $[\alpha]_D^{21}$ (hydrate) + 45·76° in H_2O (after 10 minutes). Reduces Fehling's.

Phenylhydrazone: yellow cryst. from EtOH.Aq. M.p. 146–7° (136°).

Phenylosazone: yellow needles from EtOH.Aq. M.p. 141°.

1 : 2 : 3 : 4-*Tetra-acetyl*: cryst. from EtOH. M.p. 136°.

Brigl, Zerrweck, *Z. physiol. Chem.*, 1934, **229**, 117.
Ohle, *Biochem. Z.*, 1922, **131**, 611.
Fischer, Noth, *Ber.*, 1918, **51**, 326.

***n*-Valeraldehyde** (*Pentanal, valeric aldehyde*)

$CH_3\cdot CH_2\cdot CH_2\cdot CH_2\cdot CHO$

$C_5H_{10}O$ MW, 86

Liq. B.p. 102·5–103·0°. D_4^{20} 0·80952. n_D^{20} 1·39436.

Di-Et acetal: $C_9H_{20}O_2$. MW, 160. B.p. 59°/12 mm. D^{22} 0·829. n_D^{22} 1·4029.

Oxime: m.p. 52°.

Thiosemicarbazone: cryst. from EtOH.Aq., or Et_2O. M.p. 65°.

Phenylsemicarbazone: m.p. 126–7°.

2 : 4-*Dinitrophenylhydrazone*: yellow cryst. M.p. 106–7°.

o-*Tolylsemicarbazone*: m.p. 131–2°.

m-*Tolylsemicarbazone*: m.p. 81–2°.

p-*Tolylsemicarbazone*: m.p. 157–8°.

α-*Naphthylsemicarbazone*: m.p. 124–5°.

β-*Naphthylsemicarbazone*: m.p. 134–6°.

Lieben, Rossi, *Ann.*, 1871, **159**, 70.
Bruylants, Ernould, *Chem. Abstracts*, 1932, **26**, 3232.
Blaise, *Compt. rend.*, 1904, **138**, 698.

***n*-Valeric Acid** (*Valereanic acid, butane-1-carboxylic acid, propylacetic acid*)

$CH_3\cdot CH_2\cdot CH_2\cdot CH_2\cdot COOH$

$C_5H_{10}O_2$ MW, 102

Liq. with unpleasant odour. M.p. − 34·5°. B.p. 186·35°, 96°/23 mm., 86–8°/15 mm. Sol. 27 parts H_2O at 16°. D_4^{20} 0·9387. n_α^{19} 1·4070. Heat of comb. 681·8 Cal. $k = 1{\cdot}56 \times 10^{-5}$ at 25°.

Benzylamine salt: m.p. 56–7°.

Me ester: $C_6H_{12}O_2$. MW, 116. B.p. 127·3°. D^0 0·9097.

Et ester: $C_7H_{14}O_2$. MW, 130. B.p. 144·6°/736·5 mm. D^{20} 0·8765.

Propyl ester: $C_8H_{16}O_2$. MW, 144. B.p. 167·5°. D^0 0·8888.

n-*Butyl ester*: $C_9H_{18}O_2$. MW, 158. B.p. 185·8°. D^0 0·8847.

d-sec.-*Butyl ester*: B.p. 67°/18 mm. D_4^{13} 0·8650. n_D^{20} 1·4070. $[\alpha]_D^{20}$ + 20·72°.

n-*Amyl ester*: $C_{10}H_{20}O_2$. MW, 172. B.p. 204°. D^0 0·881.

Isoamyl ester: b.p. 187–90°.

d-sec.-n-*Amyl ester*: b.p. 86°/16 mm. D^{17} 0·8631. n_D^{20} 1·4115. $[\alpha]_D^{20}$ + 16·01°

l-*Menthyl ester*: $C_{15}H_{28}O_2$. MW, 240. B.p. 141°/15 mm. D_4^{20} 0·9074. $[\alpha]_D^{20}$ − 65·55°.

p-*Bromophenacyl ester*: m.p. 64–5° (73°).

p-*Phenylphenacyl ester*: m.p. 63–63·5°.

Chloride: C_5H_9OCl. MW, 120·5. B.p. 107 10°/756 mm. D^{15} 1·0155.

Bromide: C_5H_9OBr. MW, 165. B.p. 64°/66 mm.

Amide: *n*-valeramide. $C_5H_{11}ON$. MW, 101. Plates from EtOH. M.p. 106°. Very sol. H_2O, EtOH, Et_2O.

Imide: $C_{10}H_{19}O_2N$. MW, 185. Cryst. M.p. 100°. Very sol. Et_2O. Mod. sol. hot EtOH.

Anhydride: $C_{10}H_{18}O_3$. MW, 186. B.p. 218°/754 mm., 110–11°/15 mm. D_4^{17} 0·9223.

Nitrile: *n*-valeronitrile. C_5H_9N. MW, 83. B.p. 141·1–141·2°/764·7 mm. D_4^{15} 0·80348. n_D^{15} 1·39913.

Anilide: *n*-valeranilide. Prisms from EtOH, cryst. from pet. ether. M.p. 63°.

p-*Bromoanilide*: m.p. 108°.

p-*Toluidide*: cryst. from EtOH. Plates from pet. ether. M.p. 74°.

1-*Naphthalide*: cryst. from EtOH. M.p. 112°.

Ivanoff, *Bull. soc. chim.*, 1925, **37**, 293.
Gilman, Kirby, *Organic Syntheses*, Collective Vol. I, 355.
Wolff, *Ann.*, 1881, **208**, 110.
Boudet, *Bull. soc. chim.*, 1948, 390.

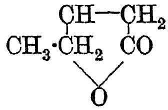
Valerolactam.

See α-Piperidone.

Valerolactinic Acid.

See 1-Hydroxy-*n*-valeric Acid.

γ-Valerolactone (3-*Methylbutyrolactone*, 3-*valerolactone*)



C_5H_8O MW, 100

dl-.

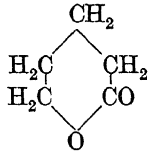
Constituent of crude pyroligneous acid. F.p. − 31° to leaflets. B.p. 205–7°, 102–3°/28 mm., 83–4°/13 mm., 78°/4 mm. Misc. in all proportions with H_2O. D^{25} 1·0465. n_D^{25} 1·4303. HI(+ P) ⟶ *n*-valeric acid. HCl ⟶ 3-chloro-*n*-valeric acid. Hyd. by $Ba(OH)_2$, slowly by H_2O. NH_3 ⟶ 3-hydroxy-*n*-valeramide.

d-.

B.p. 86–90°/14 mm. $[\alpha]_D^{20}$ + 13·5°.

Pummerer, Guyot, Birkofer, *Ber.*, 1935, **68**, 490.
Schuette, Thomas, *J. Am. Chem. Soc.*, 1930, **52**, 3011.
Levene, Haller, Walti, *J. Biol. Chem.*, 1927, **72**, 591.
Losanitsch, *Monatsh.*, 1914, **35**, 302, 311.
Boorman, Linstead, *J. Chem. Soc.*, 1933, 578.
Bissinger *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2955.

δ-Valerolactone (4-*Valerolactone*, *tetrahydro-α-pyrone*, *tetrahydrocoumalin*)



$C_5H_8O_2$ MW, 100

Oil. Solidifies on cooling. M.p. − 12·5°. B.p. 218–20°, 113–14°/13–14 mm., 88°/4 mm. Misc. with EtOH, Et_2O. Spar. misc. with H_2O. D_4^{20} 1·0794. n_D^{20} 1·4503. Readily polymerises on standing to several high MW polymers. $Ba(OH)_2$ ⟶ Ba salt of 4-hydroxy-*n*-valeric acid. HBr ⟶ 4-bromo-*n*-valeric acid.

Coffmann, *J. Am. Chem. Soc.*, 1935, **57**, 1984.
Linstead, Rydon, *J. Chem. Soc.*, 1933, 583.
Fichter, Beisswenger, *Ber.*, 1903, **36**, 1200.
Friess, *J. Am. Chem. Soc.*, 1949, **71**, 2571.

Valerone.

See Di-isobutyl Ketone.

Valerophenone (n-*Butyl phenyl ketone*)

$C_6H_5·CO·CH_2·CH_2·CH_2·CH_3$

$C_{11}H_{14}O$ MW, 162

Liq. B.p. 235–42°, 135–40°/25 mm. D_{20}^{20} 0·988. n_D^{20} 1·532.

Oxime: needles from EtOH.Aq. or pet. ether. M.p. 52·0–52·5°. B.p. 163–5°/13 mm. Sol. EtOH, C_6H_6. Spar. sol. pet. ether.

Semicarbazone: needles from EtOH. M.p. 166° (157–157·5°).

Phenylhydrazone: pale yellow viscous liq. B.p. 182°/1 mm. n_D^{17} 1·6145.

p-*Nitrophenylhydrazone*: orange cryst. from EtOH. M.p. 121–2°.

2 : 4-*Dinitrophenylhydrazone*: orange-red cryst. from EtOH. M.p. 123–4°.

Shriner, Turner, *J. Am. Chem. Soc.*, 1930, **52**, 1269.
Pfeiffer, Oberlin, *J. prakt. Chem.*, 1924, **108**, 347.
Fahim, Mustafa, *J. Chem. Soc.*, 1949, 519.

***n*-Valerylacetic Acid.**

See 2-Keto-*n*-heptylic Acid.

Valerylanisole.

See under *p*-Hydroxyvalerophenone.

4-*n*-Valerylbutyric Acid.

See 4-Ketopelargonic Acid.

Valerylcarbinol.

See 1-Hexanolone-2.

1-Valerylisopentane.

See 3-Methylnonanone-5.

Valerylphenetole.

See under *p*-Hydroxyvalerophenone.

***p*-Valerylphenol.**

See *p*-Hydroxyvalerophenone.

2-*n*-Valerylpropionic Acid.

See 3-Keto-*n*-caprylic Acid.

Validol.

See under Menthol.

Valine (1-*Aminoisovaleric acid*)

$(CH_3)_2CH·CH(NH_2)·COOH$

$C_5H_{11}O_2N$ MW, 117

d-.

Plates from EtOH.Aq. M.p. 156–157·5° (293° decomp., sealed tube). Sol. 18·4 parts H_2O at 20°. $[\alpha]_D^{20}$ − 29·04° in 20% HCl, $[\alpha]_D^{25}$ − 39·1° in Me_2CO.

Et ester: $C_7H_{15}O_2N$. MW, 145. N-p-*Nitrobenzoyl*: cryst. from ligroin. M.p. 88°. $[\alpha]_D^{20}$ − 3·5° in EtOH.

N-*Formyl*: prisms from hot H_2O. Sinters at 150°, m.p. 156°. $[\alpha]_D^{20}$ − 13·07° in EtOH, + 16·9° in H_2O.

N-p-*Toluenesulphonyl*: m.p. 149·5–150·5°. $[\alpha]_D^{22}$ − 27·7°.

Benzaldehyde deriv.: m.p. 134·5–135·5°. $[\alpha]_D^{31}$ − 51°.

l-.

Occurs in pancreas of oxen, in yellow boletus, in seeds of *Lupinus luteus* L., *Vicia sativa* L., *Lupinus angustifolius* L., etc. Plates from EtOH.Aq. M.p. 93–6° (315°, sealed tube). Sol. H_2O. Spar. sol. EtOH. $[\alpha]_D^{20}$ + 6·42° in H_2O, $[\alpha]_{5461}^{21}$ + 32·77° in HCl. $Ba(OH)_2$.Aq. at 180° ⟶ *dl*-form.

Et ester: b.p. 63·5°/8 mm. N-*Acetyl*: viscous liq. B.p. 158°/21 mm. Cryst. in plates on standing. D_4^{18} 1·028. n_D^{18} 1·4517. $[\alpha]_{5461}^{18}$ − 20·1°. N-*Benzoyl*: needles from pet. ether. M.p. 82°. $[\alpha]_D^{20}$ − 3·44° in EtOH. N-p-*Nitrobenzoyl*: needles from pet. ether. M.p. 88°. $[\alpha]_D^{20}$ + 4·12°

in EtOH. N-*Benzenesulphonyl*: cryst. from pet. ether. M.p. 56°. $[\alpha]_D^{20}$ − 1·04° in EtOH. N-p-*Toluenesulphonyl*: cryst. from pet. ether. M.p. 59°. $[\alpha]_D^{20}$ + 3·99° in EtOH.

N-*Formyl*: prisms from hot H_2O. Sinters at 150°, m.p. 156°. $[\alpha]_D^{20}$ + 13·07° in EtOH.

N-*Acetyl*: m.p. 160°. $[\alpha]_D^{19}$ + 4°.

N-*Benzoyl*: needles from H_2O. M.p. 127°. $[\alpha]_D^{20}$ + 17·18°.

N-*Benzenesulphonyl*: needles from EtOH.Aq. M.p. 153°. $[\alpha]_D^{20}$ + 19·5° in EtOH.

N-p-*Toluenesulphonyl*: needles from EtOH.Aq. M.p. 149·5–150·5°. $[\alpha]_D^{20}$ + 25·0° in EtOH.

Picrolonate: m.p. 170–80°.

dl-.

Plates from EtOH. Sublimes. M.p. about 298° decomp., (sealed tube). Sol. 11·7 parts H_2O at 15°. Insol. cold EtOH, Et_2O. Cu and Ag salts spar. sol. H_2O.

B,HCl: cryst. from hot H_2O, leaflets from dil. HCl. M.p. 189°.

Et ester: b.p. 174° part. decomp., 82·5°/23 mm., 63·5°/8 mm. D_4^{15} 0·9617. Very sol. H_2O. *B,HCl*: hygroscopic cryst. M.p. 76°. N-*Acetyl*: b.p. 99°/2 mm. *Picrate*: cryst. M.p. 139·5°.

Butyl ester: $C_9H_{19}O_2N$. MW, 173. Syrup. B.p. 98–98·5°/17 mm. D^{14} 0·9266. Sol. H_2O, EtOH, Et_2O, $CHCl_3$. *B,HCl*: needles from AcOEt. M.p. 59–60°. *Picrate*: yellow needles. M.p. 91–2°.

Amide: $C_5H_{12}ON_2$. MW, 116. Prisms from C_6H_6. M.p. 78–80°. Very sol. H_2O, EtOH, AcOEt, hot C_6H_6. Spar. sol. Et_2O, ligroin. Alk. Cu sol. ⟶ violet col. *B,HBr*: cryst. Decomp. at 200°.

Anhydride: $C_{10}H_{20}O_3N_2$. MW, 216. Does not melt below 300°.

Nitrile: $C_5H_{10}N_2$. MW, 98. Unstable yellow oil. Very sol. H_2O, EtOH, Et_2O.

N-*Me*: $C_6H_{13}O_2N$. MW, 131. Powder. Very sol. H_2O. Spar. sol. hot EtOH. Insol. Et_2O. Sublimes.

N-*Formyl*: plates from H_2O. Sinters at 137°, m.p. 140–5°.

N-*Benzoyl*: m.p. 132·5°.

p-*Iodobenzenesulphonyl*: m.p. 181·5°.

N-p-*Toluenesulphonyl*: m.p. 110–11°.

Barrow, Ferguson, *J. Chem. Soc.*, 1935, 415.
Holmes, Adams, *J. Am. Chem. Soc.*, 1934, **56**, 2093.
Karrer, Sluys Veer, *Helv. Chim. Acta*, 1932, **15**, 746.
Levene, Bass, Rothen, Steiger, *J. Biol. Chem.*, 1929, **81**, 687.
Abderhalden, Landau, *Z. physiol. Chem.*, 1911, **71**, 458.
Fischer, Scheibler, *Ber.*, 1908, **41**, 2891.
Fischer, *Ber.*, 1906, **39**, 2322.
Slimmer, *Ber.*, 1902, **35**, 401.

Valinol (2-*Aminoisoamyl alcohol*)

$(CH_3)_2CH{\cdot}CH(NH_2){\cdot}CH_2OH$

$C_5H_{13}ON$ MW, 103

D(−).

B,HCl: plates from Me_2CO. M.p. 113°. $[\alpha]_{5461}^{20}$ − 16·5° in H_2O.

Dibenzoyl deriv.: needles from EtOH.Aq. M.p. 117°. $[\alpha]_{5461}^{21}$ + 20·2° in Py.

Tribenzoyl: needles. M.p. 119°. $[\alpha]_{5461}^{20}$ − 133·8° in Py.

L(+).

Cryst. M.p. 31–2°. B.p. 88°/11 mm. $[\alpha]_D^{20}$ + 15·6° in EtOH.

B,HCl: plates from Me_2CO. M.p. 112–14° (117–18°). $[\alpha]_{5461}^{20}$ + 16·4° in H_2O, $[\alpha]_D^{20}$ + 14·25° in H_2O.

Hydrogen oxalate: m.p. 156–7°. $[\alpha]_D^{18}$ +11·25° in H_2O.

Dibenzoyl deriv.: needles. M.p. 117°. $[\alpha]_{5461}^{20}$ − 20·1° in Py.

Tribenzoyl: needles. M.p. 119°. $[\alpha]_{5461}^{19}$ + 133·1° in Py.

dl-.

Oil. B.p. 181–6°/720 mm. Sol. H_2O, EtOH. Mod. sol. Et_2O. Slowly volatile in steam.

B,HCl: plates from Me_2CO. M.p. 118–19°.

N-*Benzoyl*: needles from pet. ether. M.p. 81–2°.

Dibenzoyl deriv.: prisms from Me_2CO.Aq. M.p. 114°.

Tribenzoyl: plates from EtOH.Aq. M.p. 135°.

Barrow, Ferguson, *J. Chem. Soc.*, 1935, 410.
Karrer *et al.*, *Helv. Chim. Acta*, 1922, **5**, 478.
Karrer, Portmann, Suter, *Helv. Chim. Acta*, 1949, **32**, 1156.

Vallesine.

This name has been given to two different alkaloids from *Vallesia glabra*. The first is identical with aspidospermine (*q.v.*). The second is *N*-formyldeacetylaspidospermine, $C_{21}H_{28}O_2N_2$, MW, 340: m.p. 152–3°, $[\alpha]_D^{23}$ − 90° ± 3° in EtOH.

Hartmann, Schlittler, *Helv. Chim. Acta*, 1939, **22**, 547.
Deulofeu *et al.*, *J. Chem. Soc.*, 1940, 1051.
Schlittler, Rottenberg, *Helv. Chim. Acta*, 1948, **31**, 446.

Vanguerigenin

$C_{30}H_{46}O_3$ MW, 454

Needles + ½EtOH from EtOH. M.p. 266°. $[\alpha]_D^{22}$ + 191·3° in $CHCl_3$. Heat at 300° ⟶ vanguerol, m.p. 207°.

Me ester: cryst. from EtOH. M.p. 195°. *Acetyl*: prisms from EtOH. M.p. 248°.

Acetyl : needles + 1EtOH from EtOH. M.p. 295°.

Lactone : needles from EtOH.Aq. M.p. 281°.

Merz, Tschubel, *Ber.*, 1939, 72, 1017.

Vanguerin

$C_{41}H_{64}O_{11}$ MW, 732

From *Vangueria tomentosa*. Powder. Sinters at 255–60°. M.p. about 275–80° decomp. Sol. hot EtOH, Py. $[\alpha]_D^{22}$ − 10·1° in dioxan. Ac_2O-H_2SO_4 ⟶ yellow ⟶ red ⟶ bluish violet ⟶ brown col. Hyd. ⟶ vanguerigenin + rhamnose + arabinose.

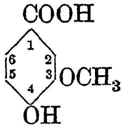
Penta-acetyl : powder. Decomp. at 184°.

Merz, Tschubel, *Ber.*, 1939, **72**, 1017.

Vanillal.

See Bourbonal.

Vanillic Acid (*Protocatechuic acid* 3-*methyl ether*, 4-*hydroxy*-3-*methoxybenzoic acid*)



$C_8H_8O_4$ MW, 168

Needles from H_2O. M.p. 210°. Sublimes. Sol. 850 parts H_2O at 14°, 62 parts at 75°, 40 parts at 95°. Sol. Et_2O. Very sol. EtOH. $k = 3{\cdot}0 \times 10^{-5}$ at 25°. Salts all very sol. H_2O. No col. with $FeCl_3$. HCl at 150° or KOH fusion ⟶ protocatechuic acid. Dist. Ca salt + Ca formate ⟶ guaiacol. Loses CO_2 with aniline at 240°.

Me ester : $C_9H_{10}O_4$. MW, 182. Needles from EtOH.Aq. M.p. 62–3°. B.p. 285–7°. *Benzoyl* : needles from MeOH. M.p. 104°.

Et ester : $C_{10}H_{12}O_4$. MW, 196. Needles. M.p. 44°. B.p. 291–3°.

Propyl ester : $C_{11}H_{14}O_4$. MW, 210. M.p. 43°.

Isopropyl ester : m.p. 113·5°.

Butyl ester : $C_{12}H_{16}O_4$. MW, 224. M.p. 48–9°.

2-*Methoxyethyl ester* : m.p. 39°. B.p. 160°/4 mm.

2-*Ethoxyethyl ester* : b.p. 142°/3 mm. n_D^{32} 1·5345. *Monohydrate* : m.p. 47–8°.

2-*Butoxyethyl ester* : b.p. 200°/3 mm. n_D^{24} 1·5161.

p-*Nitrobenzyl ester* : $C_{15}H_{13}O_6N$. MW, 303. Cryst. from EtOH.Aq. M.p. 140–1° decomp.

Chloride : $C_8H_7O_3Cl$. MW, 186·5. *Benzoyl* : needles from pet. ether. M.p. 96–8°.

Nitrile : $C_8H_7O_2N$. MW, 149. Needles from H_2O. M.p. 89–90°. Very sol. hot H_2O, EtOH, Et_2O, C_6H_6. *Acetyl* : needles from H_2O. M.p. 110°.

Me ether : *see* Veratric Acid.

Et ether : 3-methoxy-4-ethoxybenzoic acid. $C_{10}H_{12}O_4$. MW, 196. Needles from H_2O or C_6H_6. M.p. 195–6°. Sublimes. Very sol. EtOH, Et_2O. Spar. sol. hot H_2O, insol. cold.

Acetyl : needles from EtOH.Aq. M.p. 145–6°.

Benzoyl : plates from EtOH.Aq. M.p. 178°.

Sabalitsehka, Tietz, *Chem. Zentr.*, 1932, I, 1110.

Zeltner, Landau, D.R.P., 258,887, (*Chem. Zentr.*, 1913, I, 1641).

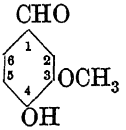
Pearl, McCoy, *J. Am. Chem., Soc.*, 1947, **69**, 3071.

Pearl, Beyer, *J. Am. Chem. Soc.*, 1949, **71**, 1066.

o-Vanillic Acid.

See under 2 : 3-Dihydroxybenzoic Acid.

Vanillin (*Protocatechuic aldehyde* 3-*methyl ether*, 4-*hydroxy*-3-*methoxybenzaldehyde*)



$C_8H_8O_3$ MW, 152

Widely occurring in plant world. Constituent of fruit of *Vanilla planifolia*, etc. Needles with characteristic odour from H_2O or ligroin. M.p. 80–1°. B.p. 170°/15 mm., 140–5°/6 mm. Very sol. EtOH, Et_2O, $CHCl_3$, CS_2, AcOH, Py. Mod. sol. hot ligroin, C_6H_6. Insol. cold ligroin. Sol. 90 parts H_2O at 14°, 20 parts at 75°. Sublimes. Heat of comb. C_p 9147 Cal., C_v 9144 Cal. Reacts in sol. as monobasic acid. Slowly oxidises in moist air ⟶ vanillic acid. KOH fusion ⟶ protocatechuic acid. NaHg or catalytic reduction ⟶ vanillyl alcohol. Electrolytic reduction ⟶ 4-hydroxy-3-methoxytoluene. $FeCl_3$ ⟶ bluish-violet col. Conc. H_2SO_4 + 1-naphthol ⟶ bluish-red col. Conc. H_2SO_4 + 2-naphthol ⟶ emerald-green col. Bromine ⟶ red col. or red ppt. Zn and Mg salts spar. sol. H_2O. Used for flavouring and in perfumery.

Me ether : *see* Veratric Aldehyde.

Et ether : $C_{10}H_{12}O_3$. MW, 180. Prisms. M.p. 64–5°. Sublimes. Sol. EtOH, Et_2O. Spar. sol. hot H_2O.

Propyl ether : $C_{11}H_{14}O_3$. MW, 194. Cryst. from EtOH.Aq. M.p. 59–60°. *Semicarbazone* : needles from EtOH. M.p. 156°.

Isopropyl ether : pale yellow viscous liq. B.p. 150–2°/13 mm. *Semicarbazone* : needles from EtOH. M.p. 151–2°.

2 : 4-*Dinitrophenyl ether* : $C_{14}H_{10}O_7N_2$. MW, 318. Needles from AcOH.Aq. M.p. 131°.

Picryl ether : $C_{14}H_9O_9N_3$. MW, 363. Plates from Et_2O–ligroin. M.p. 114–16°.

Benzyl ether : $C_{15}H_{14}O_3$. MW, 242. Needles or plates from EtOH. M.p. 64–5°.

p-*Nitrobenzyl ether* : $C_{15}H_{13}O_5N$. MW, 287. Cryst. from EtOH.Aq. M.p. 124·5°.

Acetyl : needles from Et_2O. M.p. 102–3°.

Benzoyl : needles from EtOH.Aq. M.p. 78°.

Methanesulphonyl : m.p. 89°.

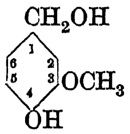
p-*Toluenesulphonyl* : needles from EtOH. M.p. about 115°.

2-*Naphthalenesulphonyl*: m.p. 98°.
β-d-*Glucoside*: m.p. 185–7°. *Tetra-acetyl*: m.p. 140–2°.
Phenylurethane: m.p. 116–17°.
Oxime: plates or needles from H_2O. M.p. 121–2°. Very sol. hot H_2O, EtOH, Et_2O. Insol. C_6H_6, ligroin. *B,HCl*: m.p. 139°. N-*Acetyl*: needles from EtOH.Aq. M.p. 114°. O:N-*Diacetyl*: pale red needles. M.p. 95°.
Azine: yellow prisms from EtOH. M.p. 195–7°, solidifies and remelts at 230–5°.
Anil: slightly yellow needles from EtOH. M.p. 152–3°.
Semicarbazone: needles. M.p. 230°.
Thiosemicarbazone: needles. Sinters at 194°, m.p. 196–7°.
Phenylhydrazone: plates from C_6H_6–ligroin. M.p. 105°.
p-*Bromophenylhydrazone*: yellow plates from EtOH. M.p. 146°.
p-*Nitrophenylhydrazone*: red plates from AcOH. M.p. 227°.
2:4-*Dinitrophenylhydrazone*: orange-red micro-prisms from AcOH. M.p. 267–8°.
Dimethone: m.p. 195·5–196·5°.

Mottern, *J. Am. Chem. Soc.*, 1934, **56**, 2107.
Hoffmann-La Roche, B.P. 399,723, (*Chem. Zentr.*, 1934, I, 127), D.R.P., 580,981, (*Chem. Zentr.*, 1933, II, 1763).

o-Vanillin.
See under 2:3-Dihydroxybenzaldehyde.

Vanillyl Alcohol (4-*Hydroxy*-3-*methoxybenzyl alcohol*)

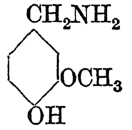


$C_8H_{10}O_3$ MW, 154

Prisms from H_2O. M.p. 115°. Not distillable at ord. press. Sol. warm H_2O, EtOH, Et_2O.
4-*Me ether*: *see* Veratryl Alcohol.
4-*Et ether*: $C_{10}H_{14}O_3$. MW, 182. Needles from EtOH. M.p. 56–7°. B.p. 185–7°/8 mm. Sol. Et_2O, EtOH, AcOEt, C_6H_6. *Allophanate*: m.p. 173°. *Acetyl*: m.p. 22–3°. B.p. 170–3°/13 mm. *Benzoyl*: m.p. 49°.
4-*Acetyl*: m.p. 51°. B.p. 194–6°/13 mm.
Diacetyl: plates from C_6H_6–ligroin. M.p. 48°. B.p. 185°/12 mm.
4-*Benzoyl*: cryst. from AcOEt–EtOH. M.p. 90° (99°).
Dibenzoyl: m.p. 121°.

Goethals, *Chem. Zentr.*, 1937, I, 580.
Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1675.
Vavon, *Ann. chim.*, 1914, **1**, 159.
Pearl, *J. Org. Chem.*, 1947, **12**, 79.

Vanillylamine (4-*Hydroxy*-3-*methoxybenzylamine*)



$C_8H_{11}O_2N$ MW, 153

Needles + $2H_2O$. M.p. anhyd. 137° (145–6°). Becomes yellow in daylight. Easily decomp. with boiling H_2O or alkalis.
B,HCl: m.p. 227°.
N-*Acetyl*: cryst. from AcOEt. M.p. 109–109·5°.
N-*Propionyl*: cryst. from C_6H_6. M.p. 111·5°.
N-*Butyryl*: cryst. from C_6H_6. M.p. 75·5–76·5°.
N-*Valeryl*: cryst. from C_6H_6–pet. ether. M.p. 60–60·5°.

Ford-Moore, Phillips, *Rec. trav. chim.*, 1934, **53**, 854.
I.G., D.R.P., 442,774, F.P., 610,835, (*Chem. Zentr.*, 1927, II, 506); U.S.P., 1,873,402, (*Chem. Abstracts*, 1932, **26**, 5965).
Jones, Pyman, *J. Chem. Soc.*, 1925, 2592.
Nelson, *J. Am. Chem. Soc.*, 1919, **41**, 1118.

Varianose

$(C_6H_{10}O_5)_n$ MW, $(162)_n$

White amorph. powder. Mod. sol. H_2O. $[\alpha]_D^{20}$ + 15° in H_2O. Reduces hot Fehling's slightly. Gives no col. with I sol. Hyd. by min. acids ⟶ *d*-galactose (70%) + *d*-glucose (14%) + either *d*-idose or *l*-altrose (14%).
Fully acetylated deriv. ("*triacetyl*"): white powder. M.p. 148–55°. Sol. Me_2CO, $CHCl_3$, hot EtOH. Does not reduce Fehling's. $[\alpha]_D^{20}$ + 30° in $CHCl_3$, + 38·2° in Me_2CO.
Fully methylated deriv. ("*trimethyl*"): white powder. M.p. 90–100°. Sol. cold, insol. hot H_2O. Sol. $CHCl_3$, Me_2CO. $[\alpha]_D^{22}$ + 15° in H_2O, + 20° in $CHCl_3$, + 23° in C_6H_6.

Haworth, Raistrick, Stacey, *Biochem. J.*, 1935, **29**, 2668.

Vasicine.
See Peganine.

Vellosine

$C_{23}H_{28}O_4N_2$ MW, 396

Constituent of bark of *Geissospermum Vellosii*. Prisms from EtOH. M.p. 189°. Sol. common org. solvents. Insol. H_2O. $[\alpha]_D$ + 22·8° in $CHCl_3$.
B,HCl: cryst. + H_2O. M.p. 180° decomp., anhyd. 245–8°.
B,HBr: needles + H_2O. M.p. 194–5°.
B,HI: plates + H_2O from EtOH.Aq. M.p. 217–18°.

B,HNO$_3$: plates + H_2O from EtOH.Aq. Decomp. at 225°.

B,H$_2$SO$_4$: cryst. + H_2O from EtOH.Aq. M.p. 210°.

B$_2$,H$_2$PtCl$_6$: cryst. M.p. about 80°.

Methiodide: cryst. from H_2O. M.p. 264°.

Freund, Fauvet, *Ann.*, 1894, **282**, 247.

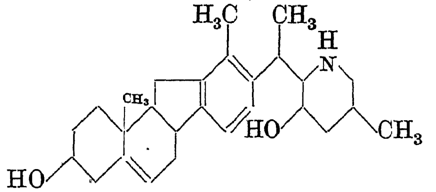
Veneniferin

$C_{32}H_{48}O_9$ MW, 576

Cardiac glycoside. Occurs in *Tanghinia venenifera*. M.p. 214°. $[\alpha]_D$ − 84° in EtOH. Hyd. ⟶ thevetose.

Frèrejacque, Hasenfratz, *Compt. rend.*, 1946, **222**, 149, 815.

Veratramine



$C_{27}H_{39}O_2N$ MW, 409

Alkaloid of white hellebore (*Veratrum grandiflorum*, Loes). Monohydrate, m.p. 209·5–210·5°. $[\alpha]_D^{19}$ anhyd. − 70° in MeOH. H(+ Pt) ⟶ dihydroveratramine, m.p. 198–200°.

B,HCl: plates from EtOH. M.p. 201–2°.

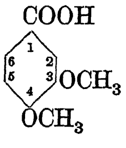
Monoacetyl: prisms from EtOH.Aq. M.p. 201–2°.

Triacetyl: needles. M.p. 205·5–206°.

Picrate: yellow plates from EtOH.Aq. M.p. 217·5–218°.

Saito, *Bull. Chem. Soc. Japan*, 1940, **15**, 22.
Jacobs, Craig, *J. Biol. Chem.*, 1945, **160**, 555.
Tamm, Wintersteiner, *J. Am. Chem. Soc.*, 1952, **74**, 3842.

Veratric Acid (*Protocatechuic acid dimethyl ether*, 3 : 4-*dimethoxybenzoic acid*)



$C_9H_{10}O_4$ MW, 182

Needles from H_2O or AcOH.Aq. M.p. anhyd. 181–2°. Sublimes in rhombic cryst. Sol. 2,100 parts H_2O at 14°, 160 parts at 100°. Very sol. EtOH, Et_2O. $k = 3{\cdot}6 \times 10^{-5}$ at 25°. Ba salt spar. sol. H_2O.

Me ester: $C_{10}H_{12}O_4$. MW, 196. Needles from EtOH.Aq. M.p. 59–60°. B.p. 300° (283°). Sol. EtOH, Et_2O. Spar. sol. H_2O.

Et ester: $C_{11}H_{14}O_4$. MW, 210. Needles from EtOH. M.p. 43–4°. B.p. 295–6°. Sol. EtOH, Et_2O. Insol. H_2O.

Chloride: $C_9H_9O_3Cl$. MW, 200·5. Cryst. M.p. 70°. B.p. about 290°.

Amide: $C_9H_{11}O_3N$. MW, 181. Cryst. M.p. 164°. Sol. hot H_2O. N-*Di-Me*: $C_{11}H_{15}O_3N$. MW, 209. M.p. 103°. B.p. 203°/12 mm. N-*Di-Et*: $C_{13}H_{19}O_3N$. MW, 237. B.p. 205°/12 mm.

Nitrile: $C_9H_9O_2N$. MW, 163. Needles from H_2O. M.p. 67–8°.

Anhydride: $C_{18}H_{18}O_7$. MW, 346. Prisms from AcOEt or C_6H_6. M.p. 124–5°.

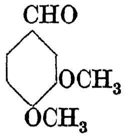
Anilide: needles from EtOH. M.p. 154°.

Buck, Ide, *Organic Syntheses*, 1935, XV, 85.
Grignard, *Compt. rend.*, 1934, **198**, 625.
Rodionov, Fedorova, *Arch. Pharm.*, 1933, **271**, 287.
Tiemann, Matsmoto, *Ber.*, 1876, **9**, 937.
Wittmer, Raiford, *J. Org. Chem.*, 1945, **10**, 527.

o-Veratric Acid.

See 2 : 3-Dimethoxybenzoic Acid.

Veratric Aldehyde (3 : 4-*Dimethoxybenzaldehyde, protocatechuic aldehyde dimethyl ether, vanillin methyl ether, veratraldehyde*)



$C_9H_{10}O_3$ MW, 166

Needles from Et_2O, ligroin, CCl_4 or toluene. M.p. 58° (42°). B.p. 285°, 154–5°/10 mm. Sol. EtOH, Et_2O. Insol. cold H_2O. Readily oxidises in air.

Oxime: cryst. from ligroin. M.p. 94–5°. Sol. common org. solvents. *B,HCl*: dark red solid. M.p. 151°.

Anil: cryst. from EtOH. M.p. 81°.

Azine: yellow plates from EtOH–$CHCl_3$. M.p. 169–70°.

Semicarbazone: m.p. 177°.

Phenylhydrazone: yellow plates from EtOH. M.p. 121°.

2 : 4-*Dinitrophenylhydrazone*: orange prisms from AcOEt. M.p. 264–5°.

Dimethone: cryst. from MeOH.Aq. M.p. 173–4°.

Johnson, Stevenson, *Organic Syntheses*, 1936, XVI, 91.
Briner, Tscharner, Paillard, *Helv. Chim. Acta*, 1925, **8**, 406.
Gatterman, *Ann.*, 1907, **357**, 367.
Weijlard, Tashjian, Tischler, *J. Am. Chem. Soc.*, 1947, **69**, 2070.

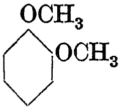
o-Veratric Aldehyde.

See under 2 : 3-Dihydroxybenzaldehyde.

Veratrine.

See Cevadine.

Veratrol (*Catechol dimethyl ether*, 1 : 2-*dimethoxybenzene*)



$C_8H_{10}O_2$ MW, 138

Cryst. from pet. ether. M.p. 22·5°. B.p. 206°/759 mm. D_{25}^{25} 1·0842. $n_{\alpha}^{21\cdot2}$ 1·52870.

Picrate : red plates. M.p. 56–7°.

Voss, Blaneke, *Ann.*, 1931, **485**, 279.
Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1649.
King, Wright, *J. Chem. Soc.*, 1939, 1168.

2-Veratroylbenzoic Acid.

See under 3′ : 4′-Dihydroxybenzophenone-2-carboxylic Acid.

Veratroylcarbinol.

See under 3 : 4-Dihydroxyphenacyl Alcohol.

2-Veratroylcinchomeronic Acid.

See Papaveric Acid.

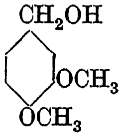
Veratroylformic Acid.

See under 3 : 4-Dihydroxybenzoylformic Acid.

Veratroyltropine.

See Convolvamine.

Veratryl Alcohol (3 : 4-*Dimethoxybenzyl alcohol*)



$C_9H_{12}O_3$ MW, 168

Viscous oil. B.p. 296–7°/732 mm., 172°/12 mm., 135°/1 mm. D_{17}^{17} 1·179. n_D^{17} 1·555. Sol. H_2O, EtOH.

Acetyl : viscous oil. B.p. 170°/12 mm. D_4^{17} 1·157. n_D^{17} 1·5245.

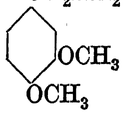
Benzoyl : m.p. 36–7°. B.p. 233–6°/12 mm. D^{13} 1·193. n_D^{13} 1·575.

p-*Nitrobenzoyl* : cryst. from MeOH. M.p. 108–9°.

Phenylurethane : m.p. 118°.

Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 905.
Tiffeneau, Fuhrer, *Bull. soc. chim.*, 1914, **15**, 171.
Vavon, *Compt. rend.*, 1912, **154**, 360.
Tiffeneau, *Bull. soc. chim.*, 1911, **9**, 929.
Mozingo, Folkers, *J. Am. Chem. Soc.*, 1948, **70**, 229.

Veratrylamine (3 : 4-*Dimethoxybenzylamine*)



$C_9H_{13}O_2N$ MW, 167

Liq. B.p. 154–8°/12 mm. D^0 1·143. Slowly volatile in steam.

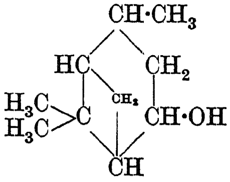
B,HCl : cryst. from EtOH. M.p. 257–8°.

Picrate : m.p. 169°.

Methiodide : m.p. 228°.

Juliusberg, *Ber.*, 1907, **40**, 120.
Nelson, *J. Am. Chem. Soc.*, 1919, **41**, 1117.
Douetteau, *Bull. soc. chim.*, 1911, **9**, 937.

Verbanol (4-*Pinanol*)

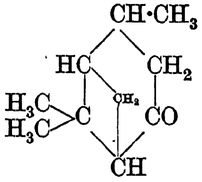


$C_{10}H_{18}O$ MW, 154

Needles from EtOH.Aq. or ligroin. M.p. 58°. B.p. 218°, 102°/20 mm. D^{20} 0·940. n_D^{20} 1·47018. $[\alpha]_D + 24·4°$.

Blumann, Zeitschel, *Ber.*, 1913, **46**, 1191.

Verbanone (4-*Pinanone*)



$C_{10}H_{16}O$ MW, 152

B.p. 222°, 117°/32 mm. D^{20} 0·961. n_D^{20} 1·47518. $[\alpha]_D + 52·5°$.

Oxime : plates from ligroin. M.p. 88° (77–8°). B.p. 148–60°/35 mm. $[\alpha]_D - 30·5°$. *B,HCl* : prisms. M.p. 142° (rapid heat.).

Semicarbazone : cryst. from EtOH.Aq. Decomp. at 230°.

Hydrazone : m.p. 27°. B.p. 146°/22 mm. D_4^{27} 0·986. n_D^{27} 1·51734. $[\alpha]_D^{27} - 29·2°$.

Wienhaus, Schumm, *Ann.*, 1924, **439**, 35.
Blumann, Zeitschel, *Ber.*, 1913, **46**, 1192.
Komppa, Klami, *Ber.*, 1937, **70**, 788.

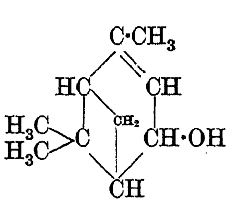
Verbascose

$C_{36}H_{62}O_{31}$ MW, 990

Occurs in roots of mullein (*Verbascum thapsus*). Needles. M.p. 219–20°. $[\alpha]_D + 169·9°$ in H_2O. Does not reduce Fehling's. Hyd. ⟶ glucose + fructose + galactose.

Bourquelot, Bridel, *Compt. rend.*, 1910, **151**, 760.

Verbenol



$C_{10}H_{16}O$ MW, 152

Cis:

M.p. 15·5°. B.p. 90°/10 mm. D^{25} 0·9684. n_D^{25} 1·4912. $[\alpha]_D$ + 65·5°.

p-*Nitrobenzoyl*: m.p. 98–9°. $[\alpha]_D$ + 114° in C_6H_6.

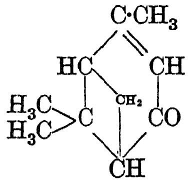
Trans:

M.p. 24°. B.p. 92°/10 mm. D^{25} 0·9657. n_D^{25} 1·4908. $[\alpha]_D$ + 168·7°.

p-*Nitrobenzoyl*: m.p. 83·5–84°. $[\alpha]_D$ + 197°.

Schulz, Doll, *Chem. Zentr.*, 1944, II, 755.

Verbenone



$C_{10}H_{14}O$ MW, 150

d-.

Constituent of Spanish verbena oil. M.p. 9·8°. B.p. 227–8°, 103–4°/16 mm. Sol. H_2O. D^{20} 0·9976. n_D^{18} 1·49928. $[\alpha]_D^{18}$ + 249·6°.

Oxime: cryst. from ligroin. M.p. 120–1°. B.p. 140–2°/17 mm. $[\alpha]_D$ + 80° in Et_2O.

Semicarbazone: plates from EtOH. M.p. 208–9°. $[\alpha]_D$ + 77·6°.

l-.

Liq. D^{15} 0·982. n_D 1·4994. $[\alpha]_D$ − 144°.

Semicarbazone: m.p. 185–90°.

dl-.

Semicarbazone: m.p. 180–1°.

Dupont, Zacharewicz, *Bull. soc. chim.*, 1935, **2**, 533.
Wienhaus, Schumm, *Ann.*, 1924, **439**, 20.
Blumann, Zeitschel, *Ber.*, 1913, **46**, 1188.
Kerschbaum, *Ber.*, 1900, **33**, 885.
Schulz, Doll, *Chem. Zentr.*, 1944, II, 755.

Vernine.

See Guanosine.

Vernolic Acid

$CH_3{\cdot}[CH_2]_6{\cdot}CH{:}CH{\cdot}CH(OH){\cdot}[CH_2]_7{\cdot}COOH$

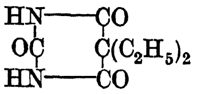
Suggested structure

$C_{18}H_{34}O_2$ MW, 282

Constituent of the seed oil of *Vermonia anthelmintica*. M.p. 21–2°.

Vidyarthi, *Chem. Abstracts*, 1946, **40**, 4899.

Veronal (5 : 5-*Diethylbarbituric acid, diethylmalonylurea, Barbitone*)



$C_8H_{12}O_3N_2$ MW, 184

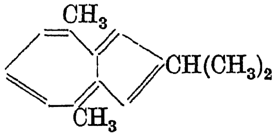
Exists in polymorphic forms. Triagonal cryst., m.p. 190°: monoclinic prisms, m.p. 183°: monoclinic needles, m.p. 181: triclinic cryst., m.p. 176°. Sol. EtOH, Et_2O, aq. alkalis. Spar. sol. $CHCl_3$. Prac. insol. H_2O. $k = 3{\cdot}7 \times 10^{-8}$ at 25°. Powerful hypnotic.

Fischer, Dilthey, *Ann.*, 1904, **335**, 338.
Köfler, *Chem. Abstracts*, 1947, **41**, 3584.

Vetivalene.

See 1 : 5-Dimethyl-3-isopropylnaphthalene.

Vetivazulene (4 : 8-*Dimethyl-2-isopropylazulene*)



$C_{15}H_{18}$ MW, 198

M.p. 31·5–32°. B.p. 140–60°/2 mm.

Picrate: black needles from EtOH. M.p. 122·5°.

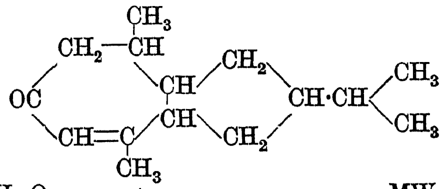
sym-Trinitrobenzene add. comp.: black needles with copper reflex from EtOH. M.p. 154–5°.

2 : 4 : 6-*Trinitrotoluene add. comp.*: copper coloured needles. M.p. 80·5–81°.

Styphnate: m.p. 95–96·7°.

Pfau, Plattner, *Helv. Chim. Acta*, 1939, **22**, 202; 1936, **19**, 858.
Coates, Cook, *J. Chem. Soc.*, 1942, 559.

β-**Vetivone** (β-*Vetiverone*)



$C_{15}H_{24}O$ MW, 220

Isolated from vetiver oil. M.p. 44–44·5°. B.p. 141–2°/2 mm. D_4^{20} 1·0001. n_D^{20} 1·5309. $[\alpha]_D$ − 38·9° in EtOH.

Semicarbazone: leaflets from Py. M.p. 228–9°. $[\alpha]_D$ − 71·1° in AcOH.

2 : 4-*Dinitrophenylhydrazone*: m.p. 190·5–191°.

Naves, Perrotet, *Helv. Chim. Acta*, 1941, **24**, 3.
Pfau, Plattner, *Helv. Chim. Acta*, 1940, **23**, 768; 1939, **22**, 640.

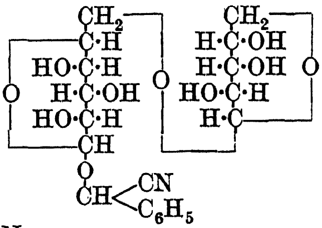
Viboquercitol.

See under Quercitol.

Viburnitol.

See under Quercitol.

Vicianin

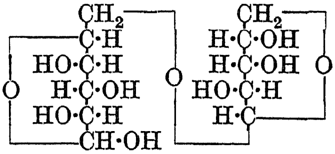


$C_{19}H_{25}O_{10}N$ MW, 427

Occurs in seeds of *Vicia angustifolia*. Needles. M.p. 147–8°. Sol. hot H_2O. Spar. sol. EtOH. Insol. $CHCl_3$, C_6H_6, CS_2, pet. ether. $[\alpha]_D$ −20·7° in H_2O. Hyd. by vicianase ⟶ vicianose + HCN + C_6H_5·CHO.

Bertrand, *Compt. rend.*, 1906, **143**, 832.
Bertrand, Weisweiller, *Compt. rend.*, 1908, **147**, 252; 1910, **151**, 884; *Bull. soc. chim.*, 1911, **9**, 147.

Vicianose



$C_{11}H_{20}O_{10}$ MW, 312

Does not occur in free state but in various glycosides, *e.g.* gein from *Geum urbanum*, vicianin from *Vicia angustifolia*, violutin from *Viola cornuta*. Needles from dil. EtOH. M.p. 210° decomp. Sinters at 190°. Sol. H_2O, dil. EtOH. Sweet taste. $[\alpha]_D^{20}$ + 56·5° ⟶ + 39·72° in H_2O. Does not ferment. Hyd. by dil. acids ⟶ *d*-glucose + *l*-arabinose.

β-*Hepta-acetyl*: needles from EtOH. M.p. 158–60°. $[\alpha]_D^{14}$ + 7·5° in $CHCl_3$. Sol. $CHCl_3$, Et_2O, Me_2CO. Spar. sol. EtOH. Prac. insol. H_2O.

Bertrand, Weisweiller, *Bull. soc. chim.*, 1911, **9**, 38, 84, 147.
Herissey, Cheymol, *Compt. rend.*, 1925, **181**, 565.
Helferich, Bredereck, *Ann.*, 1928, **465**, 166.

Vinaconic Acid.
See Cyclopropane-1 : 1-dicarboxylic Acid.

Vinopyrin.
See under p-Phenetidine.

Vinylacetic Acid (*Propylene*-3-*carboxylic acid*, 2-*methylenepropionic acid*)

CH_2:CH·CH_2·COOH

$C_4H_6O_2$ MW, 86

M.p. − 39°. B.p. 163°, 69–70°/12 mm. D_{15}^{15} 1·013. n_D^{15} 1·42572. $k = 3{\cdot}83 \times 10^{-5}$ at 25°.

Et ester: $C_6H_{10}O_2$. MW, 114. B.p. 119°. D_4^{20} 0·91224. n_D^{20} 1·41054.

Amide: C_4H_7ON. MW, 85. Plates from pet. ether. M.p. 72–3°.

Nitrile: *see* Allyl cyanide.

Anilide: needles from EtOH.Aq. M.p. 58°.

Mannich, *Arch. Pharm.*, 1935, **273**, 415.
Linstead, Noble, Boorman, *J. Chem. Soc.*, 1933, 560.
Houben, *Ber.*, 1903, **36**, 2897.
Rietz, *Organic Syntheses*, 1944, XXIV, 96.

Vinylacetone.
See Methyl allyl Ketone.

Vinylacetylacetylene.
See 1-Hexen-3-yn-5-one.

Vinylacetylene (3-*Butenyne*-1)

CH_2:CH·C⋮CH

C_4H_4 MW, 52

Gas with odour resembling acetylene. Sets to cryst. in liq. air. B.p. 2–3°. Decomp. violently on heating. Explodes with fuming HNO_3.

Shaworonkow, *Chem. Zentr.*, 1937, I, 1668.
du Pont, F.P., 43,351, addn. to F.P., 733,663, and 41,454, (*Chem. Abstracts*, 1934, **28**, 6727).
Willstätter, Wirth, *Ber.*, 1913, **46**, 538.
Nieuwland, *Ind. Eng. Chem.*, 1935, **27**, 850 (*Review*).

2-Vinylacrylic Acid (1 : 3-*Butadiene*-1-*carboxylic acid*, *erythrene*-1-*carboxylic acid*)

CH_2:CH·CH:CH·COOH

$C_5H_6O_2$ MW, 98

Prisms from Et_2O. M.p. 80°. Very hygroscopic. Very sol. Et_2O, EtOH, hot H_2O, C_6H_6. Sol. pet. ether.

Et ester: $C_7H_{10}O_2$. MW, 126. B.p. 70–1°/31 mm., 57·2–57·5°/13 mm. $D_4^{23.3}$ 0·9348. $n_\alpha^{19.8}$ 1·49306.

Nitrile: *see* Cyanoprene.

Coffmann, *J. Am. Chem. Soc.*, 1935, **57**, 1982.
Muskat, Becker, Lowenstein, *J. Am. Chem. Soc.*, 1930, **52**, 329.
Burton, Ingold, *J. Chem. Soc.*, 1929, 2028.
Noffbohm, *Ann.*, 1917, **412**, 73.

Vinyl Alcohol (*Ethenol*)

CH_2:CHOH

C_2H_4O MW, 44

Does not exist in free state. Polymers of the alcohol and its esters used in synthetic resins.

Acetyl: vinyl acetate. Polymerises in air to transparent mass, polyvinyl acetate. Used in lacquers and resins.

Benzoyl: b.p. 203°. D^{20} 1·065. Polymers used in lacquers.

Et ether: *see* Ethyl vinyl Ether.

Vinyl ether: *see* Divinyl Ether.

Phenyl ether: C_8H_8O. MW, 120. B.p. 158–60°.

I.G., D.R.P., 604,640, (*Chem. Zentr.*, 1935, II, 2125).
Evans, Looker, *J. Am. Chem. Soc.*, 1921, **43**, 1925.
Chem. Fabr. Griesheim-Elektron, D.R.P., 271,381, (*Chem. Zentr.*, 1914, I, 1316).

Vinylallylcarbinol (1 : 5-*Hexadienol*-3)

CH_2:CH·CH_2·CH(OH)·CH:CH_2

$C_6H_{10}O$ MW, 98

Liq. B.p. 133–4°, 42°/12 mm. D_4^{25} 0·8596. n_D^{25} 1·4464.

Levene, Haller, *J. Biol. Chem.*, 1929, **83**, 185.

Butz, Butz, Gaddis, *J. Org. Chem.*, 1940, **5**, 171.

***sym.*-Vinylallylethylene.**
See 1 : 3 : 6-Heptatriene.

Vinylamine.
See Ethyleneimine.

Vinylanisole.
See under Hydroxystyrene.

Vinylbenzene.
See Styrene.

Vinyl bromide (*Bromoethylene*)

$CH_2{:}CHBr$

C_2H_3Br MW, 107

F.p. − 137·8°. B.p. 15·8°. D_4^{11} 1·5286. n_D 1·4462. H_2O + PbO ⟶ acetylene. $KMnO_4$ ⟶ H·COOH. Polymerises rapidly in sunlight ⟶ mixture of polymers.

Staudinger, Brunner, Feisst, *Helv. Chim. Acta*, 1930, **13**, 805.

Juvala, *Ber.*, 1930, **63**, 1991.

Bauer, U.S.P., 1,414,852, (*Chem. Abstracts*, 1922, **16**, 2150).

2-Vinylbutane.
See 3-Methyl-1-pentene.

Vinyl-*n*-butyl Alcohol.
See 1-Hexenol-6.

Vinylbutylene.
See 1 : 3-Hexadiene, 1 : 4-Hexadiene *and* 3-Methyl-1 : 3-pentadiene.

Vinyl chloride (*Chloroethylene*)

$CH_2{:}CHCl$

C_2H_3Cl MW, 62·5

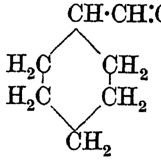
Gas. Liquefies on cooling. F.p. − 159·7°. B.p. − 14°. Heat of comb. C_p 286·2 Cal. Polymerises in light or in absence of light in presence of catalysts.

Carbide and Carbon Chem. Corp., U.S.P., 1,934,824, (*Chem. Abstracts*, 1934, **28**, 488); U.S.P., 1,926,638, (*Chem. Abstracts*, 1933, **27**, 5756).

Dana, Burdick, Jenkins, *J. Am. Chem. Soc.*, 1927, **49**, 2801.

Vinyl cyanide.
See under Acrylic Acid.

Vinylcyclohexane (*Cyclohexylethylene*)

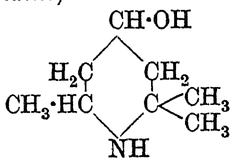


C_8H_{14} MW, 110

Liq. B.p. 130–1°/749 mm. D_4^{19} 0·8166. n_D^{19} 1·455.

Lewina, Zurikow, *Chem. Zentr.*, 1936, I, 4289.

Vinyldiacetonalkamine (4-*Hydroxy*-2 : 2 : 6-*trimethylpiperidine*)



$C_8H_{17}ON$ MW, 143

α-Form :

d-.

B,HCl : does not melt below 300°. $[\alpha]_D$ + 13·3° in H_2O.

l-.

Prisms from C_6H_6. M.p. 79–81°.

B,HCl : does not melt below 300°. $[\alpha]_{5461}$ − 18·5° in H_2O.

Picrate : needles. M.p. 242–4°.

dl-.

Cryst. from C_6H_6. M.p. 137–8°. B.p. 209–11°. Sol. 9 parts boiling H_2O. Spar. sol. pet. ether.

B,HCl : prisms from EtOH.Aq.

N-*Me* : $C_9H_{19}ON$. MW, 157. Pale yellow oil. B.p. 218–20°. Very sol. H_2O. *Dihydrate* : plates from C_6H_6–pet. ether. M.p. 39–40°. *B,HCl* : m.p. 192°. *B_2,H_2PtCl_6* : cryst. M.p. 208° decomp. O-*Benzoyl* : pale yellow oil. B.p. 194–5°/16 mm. *Picrate* : cryst. from EtOH.Aq. M.p. 180–1°.

N-*Nitroso* : $C_8H_{16}O_2N_2$. MW, 172. Cryst. from C_6H_6–pet. ether. M.p. 92°.

O-*Benzoyl* : *see* β-Eucaine.

Picrate : plates from H_2O. M.p. 171–2°.

β-Form :

d-.

Plates from C_6H_6. M.p. 121–3°.

B,HCl : plates. M.p. 217–19°. $[\alpha]_{5461}$ + 22·85°.

Picrate : needles from H_2O. M.p. 181–2°.

dl-.

Prisms from C_6H_6. M.p. 160–1°. B.p. 204–5°. Sol. 30 parts boiling C_6H_6. Spar. sol. H_2O, Et_2O. Na amylate in amyl alcohol ⟶ *dl*-α-form.

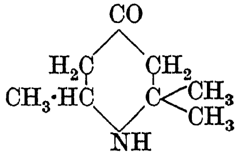
N-*Me* : prisms from pet. ether. M.p. 70–2°. B.p. 220°/744 mm. Very sol. H_2O. Na amylate in amyl alcohol ⟶ *dl*-α-*N*-Me. *B,HCl* : m.p. 58°. *Nitrate* : prisms. M.p. 163° decomp. *B_2,H_2PtCl_6* : cryst. M.p. 218° decomp. *Methiodide* : prisms from EtOH. Decomp. at 270°. O-*Benzoyl* : pale yellow oil. B.p. 195°/15 mm. *Picrate* : yellow cryst. from EtOH.Aq. M.p. 213°.

N-*Nitroso* : yellow needles from C_6H_6–pet. ether. M.p. 60°.

O-*Benzoyl*: *see* Iso-β-eucaine.
Picrate: plates from H_2O. M.p. 171–2°.

King, *J. Chem. Soc.*, 1924, **125**, 41.
Harries, Zart, *Ann.*, 1918, **417**, 176.
Harries, *Ann.*, 1897, **294**, 372; 1897, **296**, 334.

Vinyldiacetonamine (4-*Keto*-2 : 2 : 6-*trimethylpiperidine*, 2 : 2 : 6-*trimethyl-γ-piperidone*)



$C_8H_{15}ON$ MW, 141

Hygroscopic plates and prisms. M.p. 27°. B.p. 199–200°. NaHg ⟶ vinyldiacetonalkamine.

B_2,H_2SO_4: needles from EtOH.Aq. M.p. above 105° decomp.
Nitrate: needles from Me_2CO–pet. ether. M.p. 160–1°.
$B,(COOH)_2$: m.p. 184–5° decomp.
N-*Me*: $C_9H_{17}ON$. MW, 155. Oil. B.p. 96–7°/14 mm. *Oxime*: prisms from pet. ether. M.p. 93°.
N-*Nitroso*: $C_8H_{14}O_2N_2$. MW, 170. Pale yellow plates from MeOH.Aq. M.p. 61°. *Oxime*: cryst. from H_2O. M.p. 195°.
N-*Acetyl*: prisms from Et_2O. M.p. 92°.
N-m-*Nitrobenzoyl*: needles from Me_2CO. M.p. 159–60°.
N-p-*Nitrobenzoyl*: needles from Me_2CO. M.p. 170°.
Oxime: plates from EtOH. M.p. 151°. Sol. 5 parts boiling H_2O. N-*Acetyl*: cryst. from EtOH. M.p. 130°.
Semicarbazone: cryst. from EtOH–C_6H_6. M.p. 196–7°.
Picrate: prisms from H_2O. M.p. 198–9°.

Harries, Schellhorn, *Ann.*, 1918, **417**, 131.
Harries, *Ber.*, 1896, **29**, 522.

α-Vinyldiphenylmethane.
See 3 : 3-Diphenylpropylene.

Vinylethynylcarbinol.
See 4-Penten-1-yn-3-ol.

Vinyl fluoride (*Fluoroethylene*)

CH_2:CHF

C_2H_3F MW, 46

Gas at ord. temps. B.p. — 51°. Burns with green flame. Sol. EtOH, Me_2CO. Insol. H_2O.

Swarts, *Bull. soc. chim.*, 1919, **25**, 163; *Chem. Zentr.*, 1909, II, 1414; 1901, II, 804.
Newkirk, *J. Am. Chem. Soc.*, 1946, **68**, 2467.

Vinylformic Acid.
See Acrylic Acid.

Vinylfuran.
See α-Furylethylene.

Vinylglycollic Acid (1-*Hydroxyvinylacetic acid*)

CH_2:CH·CH(OH)·COOH

$C_4H_6O_3$ MW, 102

Hygroscopic needles. M.p. 42–4°. B.p. 128·6–130·2°/12–13 mm. Very sol. H_2O. Sol. EtOH, Et_2O, $CHCl_3$. Insol. CS_2. Volatile in steam. $k = 4{\cdot}6 \times 10^{-4}$. Min. acids ⟶ 1-ketobutyric acid.

Et ester: $C_6H_{10}O_3$. MW, 130. Liq. with pleasant odour. B.p. 173° slight decomp., 68°/15 mm. D_4^{15} 1·0470. n_D^{13} 1·436. *Acetyl*: b.p. 89°/15 mm. D^{16} 1·055. n_D^{16} 1·429.
Amide: $C_4H_7O_2N$. MW, 101. Plates from Et_2O. M.p. 80·8°. B.p. 155–8°/20–1 mm. slight decomp. Sol. H_2O, EtOH. Spar. sol. Et_2O.
Nitrile: *see under* Acrolein.

Glattfield, Hoen, *J. Am. Chem. Soc.*, 1935, **57**, 1406.
Kirrmann, Rambaud, *Compt. rend.*, 1932, **194**, 1169.
Sleen, *Rec. trav. chim.*, 1902, **21**, 222.

Vinylidene bromide.
See unsym.-Dibromoethylene.

Vinylidene chloride.
See unsym.-Dichloroethylene.

Vinylidene-ethyl Alcohol.
See 4-Hydroxy-1 : 2-butadiene.

Vinylidene fluoride.
See Difluoroethylene.

Vinyl iodide (*Iodoethylene*)

CH_2:CHI

C_2H_3I MW, 154

B.p. 56–56·5°. D^{20} 2·037. n_D 1·53845. Decomp. on exposure to ultra-violet light ⟶ C_2H_2, C_2H_4 and I.

Spence, *J. Am. Chem. Soc.*, 1933, **55**, 1290.

Vinylisoamylcarbinol.
See 6-Methyl-1-heptenol-3.

1-Vinylisobutylene.
See 4-Methyl-1 : 3-pentadiene.

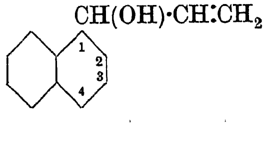
Vinylisobutyric Acid.
See Dimethylvinylacetic Acid.

1-Vinylisopentane.
See 4-Methyl-1-hexene.

***sym*.-Vinylisopropenylethane.**
See 2-Methyl-1 : 5-hexadiene.

Vinylnaphthalene.
See Naphthylethylene.

Vinyl-1-naphthylcarbinol (1-α-*Naphthylallyl alcohol*, 1-α-*hydroxyallylnaphthalene*)



$C_{13}H_{12}O$ MW, 184

Viscous oil. B.p. 186–7°/19 mm.

Phenylurethane: needles from pet. ether. M.p. 108–9°.

p-*Nitrobenzoyl*: prisms from EtOH. M.p. 79–80°.

Burton, *J. Chem. Soc.*, 1931, 761.

Vinyl-2-naphthylcarbinol (1-β-*Naphthylallyl alcohol, 2-α-hydroxyallylnaphthalene*).

Viscous oil. B.p. 195–8°/21 mm.

Phenylurethane: prisms from C_6H_6–pet. ether. M.p. 134–5°.

Burton, *J. Chem. Soc.*, 1931, 761.

2-Vinylpentane.

See 3-Methyl-1-hexene.

1-Vinyl-2-pentene.

See 1 : 4-Heptadiene.

Vinylphenetole.

See under p-Hydroxystyrene.

Vinylphenol.

See Hydroxystyrene.

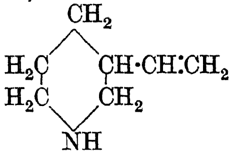
Vinylphenylcarbinol.

See 1-Phenylallyl Alcohol.

Vinyl phenyl Ketone.

See Acrylophenone.

3-Vinylpiperidine (β-*Vinylpiperidine, 3-piperidylethylene*)



$C_7H_{13}N$ MW, 111

Liq. B.p. 152–5°. D_4^{25} 0·9274. n_D^{25} 1·4731.

B_2,H_2PtCl_6: cryst. + $2H_2O$ from EtOH.Aq. M.p. 223–4° decomp.

N-*Me*: $C_8H_{15}N$. MW, 125. Liq. B.p. 161–2°/724 mm. Sol. 30 parts cold H_2O. Spar. sol. hot H_2O. Decolorises acid $KMnO_4$. Zn + HCl ⟶ 1-methyl-3-ethylpiperidine. $B,HAuCl_4$: prisms from H_2O. M.p. 58–9°. B_2,H_2PtCl_6: orange-red plates or prisms. M.p. 185–6° decomp. *Picrate*: yellow prisms or needles. M.p. 193–4°.

N-*Et*: $C_9H_{17}N$. MW, 139. Liq. B.p. 185–90°/721 mm. Spar. sol. H_2O. $B,HAuCl_4$: cryst. M.p. 44–5°. B_2,H_2PtCl_6: cryst. from H_2O. M.p. 175–6° decomp.

Picrate: m.p. 162–4°.

Merchant, Marvel, *J. Am. Chem. Soc.*, 1928, **50**, 1201.

Lipp, Widmann, *Ann.*, 1915, **409**, 92; *Ber.*, 1905, **38**, 2481.

Iddles, Lang, Gregg, *J. Am. Chem. Soc.*, 1937, **59**, 1945.

4-[3-Vinylpiperidyl]-acetic Acid.

See Meroquinene.

2-Vinylpropane.

See 3-Methyl-1-butylene.

***sym*.-Vinylpropenylethylene.**

See 1 : 3 : 5-Heptatriene.

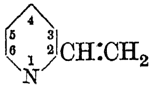
2-Vinylpropionic Acid.

See Allylacetic Acid.

1-Vinyl-3-propylidene-propylene.

See 1 : 3 : 5-Heptatriene.

2-Vinylpyridine (α-*Vinylpyridine, 2-pyridylethylene*)



C_7H_7N MW, 105

Liq. B.p. 79–82°/29 mm. D^0 0·9985. Very sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. H_2O. Volatile in steam. $KMnO_4$ ⟶ picolinic acid. Na + EtOH ⟶ 2-ethylpiperidine.

$B,HAuCl_4$: yellow needles from H_2O. M.p. 144°.

B_2,H_2PtCl_6: needles or plates. M.p. 174° decomp.

Löffler, Grosse, *Ber.*, 1907, **40**, 1326.

3-Vinylpyridine (β-*Vinylpyridine, 3-pyridylethylene*).

Unstable light yellow liq. Polymerises at room temp. Sol. Et_2O. Part. sol. EtOH. Spar. sol. H_2O.

B,HCl: cryst. from EtOH. M.p. 114–15°. Insol. cold EtOH, Et_2O, H_2O.

$B,HAuCl_4$: yellow cryst. from EtOH. M.p. 138–40°.

B_2,H_2PtCl_6: light orange cryst. from EtOH. M.p. 158–60°.

$B,HgCl_2$: cryst. from EtOH. M.p. 145–50°.

Picrate: yellow cryst. from C_6H_6. M.p. 143–4°. Insol. EtOH, Et_2O, H_2O.

Iddles, Lang, Gregg, *J. Am. Chem. Soc.*, 1937, **59**, 1945.

4-Vinylpyridine (γ-*Vinylpyridine, 4-pyridylethylene*).

Liq. B.p. 59°/12 mm., 54°/7 mm. n_D^{25} 1·5490.

B,H_2PtCl_3: sinters at 200°, m.p. above 350°.

Picrate: yellow leaflets from C_6H_6. M.p. 197–8°.

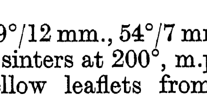
Meisenheimer, *Ann.*, 1920, **420**, 208.

Brown, Eldred, *J. Am. Chem. Soc.*, 1949, **71**, 445.

Vinylpyridinium hydroxide.

See Pyridine-neurine.

2-Vinylquinoline (α-*Vinylquinoline, 2-quinolylethylene*)



$C_{11}H_9N$ MW, 155

Pale yellow oil. Decomp. on dist. at 10 mm. Volatile in steam.

$B,HgCl_2,HCl$: needles from H_2O. M.p. 151–2° decomp.

$B,HAuCl_4$: yellowish-red needles from dil. min. acids. M.p. 158–9°.

B_2,H_2PtCl_6: orange-red needles. Decomp. at 182°.

B_2,H_2PtCl_6,$4H_2O$: orange needles from dil. min. acids. M.p. 186–7°.

Methner, *Ber.*, 1894, **27**, 2691.

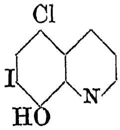
Vinyl sulphide.
See Divinyl sulphide.

Vinyltoluene.
See Methylstyrene.

4-Vinylvaleric Acid.
See 5-Heptenic Acid.

Vinylxylene.
See Dimethylstyrene.

Vioform (*Nioform, 5-chloro-7-iodo-8-hydroxyquinoline*)



C_9H_5ONClI MW, 305·5

M.p. 178–9°. Used in treatment of amœbic enteritis.

Sen-Gupta, *J. Indian. Chem. Soc.*, 1945, **22**, 171.

Violacein

$C_{42}H_{28}O_7N_6$? MW, 728 ?

A violet phenolic pigment produced by *Chromobacterium violaceum*. Appears to have eight double bonds and to contain pyrrole nuclei.

Beer *et al.*, *J. Chem. Soc.*, 1949, 885.

Violanin (*Rhamnoglucoside of delphinidin*)

$C_{27}H_{28}O_{15}$ MW, 592

Constituent of *Viola tricolor*, Linn.

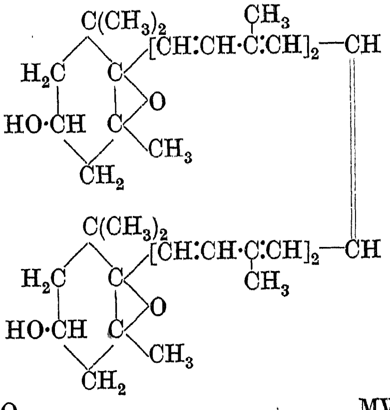
Chloride: bluish-violet plates with green metallic lustre. Sol. alkalis and alk. carbonates ⟶ blue sols. Alc. $FeCl_3$ ⟶ blue col. Boiling dil. HCl ⟶ delphinidin + rhamnose + glucose.

Willstätter, Weil, *Ann.*, 1916, **412**, 178.

Violanthrone.
See Dibenzanthrone.

Violaquercitrin.
See Rutin.

Violaxanthin



$C_{40}H_{56}O_4$ MW, 600

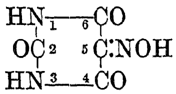
Constituent of *Viola tricoloris*, etc. Red prisms from MeOH, EtOH–Et_2O, or CS_2. M.p. 208° (198–9°). $[\alpha]^{20}_{\mathrm{Cd}}$ + 35° (+ 38°) in $CHCl_3$. Optical maxima in CS_2 at 500·5, 469 and 440 mμ, in $CHCl_3$ at 482, 451·5 and 424 mμ. Sol. conc. H_2SO_4 ⟶ indigo-blue col. Sol. H·COOH ⟶ blue col. Sol. cold AcOH ⟶ green col. $SbCl_3$ + $CHCl_3$ ⟶ deep blue col. Conc. HCl in AcOH ⟶ greenish-blue col.

Dibenzoyl: cryst. from C_6H_6–MeOH. M.p. 217°.

Di-p-*nitrobenzoyl*: cryst. from C_6H_6. M.p. 208°.

Zechmeister, Tuzson, *Ber.*, 1934, **67**, 824.
Kuhn, Grundmann, *ibid.*, 596.
Zechmeister, Cholnoky, *Z. physiol. Chem.*, 1932, **208**, 26.
Kuhn, Winterstein, *Ber.*, 1931, **64**, 326.
Karrer, Morf, *Helv. Chim. Acta*, 1931, **14**, 1044.
Karrer, Jucker, *Helv. Chim. Acta*, 1945, **28**, 300.

Violuric Acid (*5-Isonitrosobarbituric acid, alloxan-5-oxime*)



$C_4H_3O_4N_3$ MW, 157

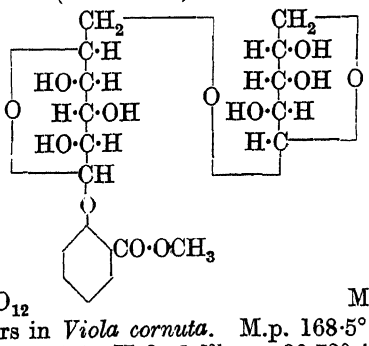
Rhombic cryst. Mod. sol. H_2O ⟶ violet sol. Sol. EtOH. $k = 2{\cdot}7 \times 10^{-5}$ at 25°. $FeCl_3$ ⟶ blue col. Warm with HCl ⟶ NH_2OH. Red. ⟶ uramil. Forms series of variously coloured metallic salts.

5-O-*Me ether*: $C_5H_5O_4N_3$. MW, 171. Plates from ligroin. M.p. 268°. Sol. H_2O, alkalis and org. solvents ⟶ yellow sols. $k = 1{\cdot}8 \times 10^{-7}$ at 25°.

5-O-*Benzyl ether*: $C_{11}H_9O_4N_3$. MW, 247. Plates from C_6H_6. M.p. 226° decomp. Sol. H_2O, EtOH.

Andreasch, *Monatsh.*, 1900, **21**, 286.
Hantzsch, Isherwood, *Ber.*, 1909, **42**, 986.

Violutin (*Violutoside*)



$C_{19}H_{26}O_{12}$ MW, 446

Occurs in *Viola cornuta*. M.p. 168·5° (173°). $[\alpha]_D$ − 36·20° in H_2O, $[\alpha]^{21}_{5461}$ − 39·72° in H_2O, $[\alpha]^{21}_{5790}$ − 36·21° in H_2O. Sol. H_2O. Prac. insol. dry Me_2CO or AcOEt. Hyd. ⟶ methyl salicylate + *d*-glucose + *l*-arabinose.

Hexa-acetyl: m.p. 158°. $[\alpha]_{5461}^{21}$ − 42·94° in Me_2CO.

Picard, *Compt. rend.*, 1926, **182**, 1167.
Robertson, Waters, *J. Chem. Soc.*, 1932, 2770.

Violutoside.
See Violutin.

Viomycin

$C_{18}H_{31-33}O_8N_9$ MW, 501 (503)

A tuberculostatic antibiotic obtained from *Streptomyces puniceus* and *Streptomyces floridæ*. A strongly basic polypeptide. Acid hyd. ⟶ CO_2, NH_3, urea, L-serine, αβ-diaminopropionic acid, and a basic amino acid isomeric with lysine.

Haskell *et al.*, *J. Am. Chem. Soc.*, 1952, **74**, 599.

Viridin

$C_{19}H_{16}O_6$ MW, 340

A highly fungistatic substance produced by *Trichoderma viride*. Has been separated into two isomers.

α-.
Cryst. Decomp. at 208–17°. $[\alpha]_D^{20}$ −214·4° in $CHCl_3$. $\lambda_{max.}$ 2413 Å, ε = 31,000 in EtOH; $\lambda_{max.}$ 3045 Å, ε = 13,200 in EtOH.
Diacetyl deriv.: m.p. 145–50°.

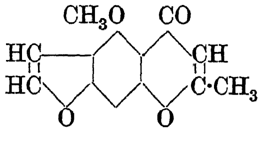
β-.
Needles from MeOH. M.p. 140°. $[\alpha]_D^{20}$ + 50·7° in $CHCl_3$.

Brian, McGowan, *Nature*, 1945, **156**, 144.
Vischer, Howland, Raudnitz, *Nature*, 1950, **165**, 528.

α-Viscol.
See β-Amyrin.

β-Viscol.
See Lupeol.

Visnagin (*5-Methoxy-2-methylfuro*-[3′,2′-6,7]-*chromone*)



$C_{13}H_{10}O_4$ MW, 230

Isolated from seeds of *Ammi visnaga*. Cryst. from MeOH. M.p. 144–5°.

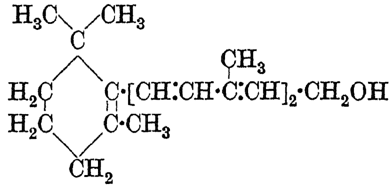
Späth, Gruber, *Ber.*, 1941, **74**, 1492.
Geissman, Hinreiner, *J. Am. Chem. Soc.*, 1951, **73**, 782.
Gruber, Horváth, *Monatsh*, 1949, **80**, 874.

Visual Purple (*Rhodopsin*).

One of several photo-labile pigments of the retinal rods and cones. A conjugated rose-coloured protein, vitamin A being the prosthetic group. The pigment is responsible for scotopic vision, although not for photopic vision.

Collins, Morton, *Biochem. J.*, 1950, **47**, 3, 10, 18.
Wald, Brown, *Proceedings of the National Academy of Science, U.S.A.*, 1950, **36**, 84.

Vitamin A_1 (*Antixerophthalmic, anti-infective and growth-promoting vitamin*)

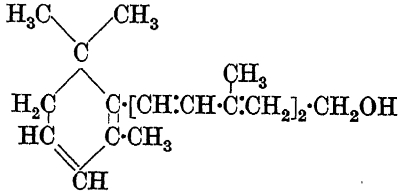


$C_{20}H_{30}O$ MW, 286

Constituent of many fish-liver oils, milk, egg-yolk, seed embryos, etc. Yellow cryst. M.p. 60–2°. B.p. 137–8°/10^{-6} mm. Sol. all org. solvents. Insol. H_2O. Unstable in air. Characteristic absorption band at 328 mμ. $SbCl_3$ in $CHCl_3$ ⟶ blue sol. with absorption bands at 583 and 617 mμ. Conc. H_2SO_4 ⟶ violet col. Physiological activity destroyed on reduction.
2-Naphthoyl: yellow plates from EtOH. M.p. 74–5°.
Anthraquinone-2-carboxylate: pale yellow prisms from Me_2CO. M.p. 124°. Red modification, m.p. 117–19°.
p-*Phenylazobenzoyl*: orange-yellow plates. M.p. 79–80°.

Karrer, Morf, Schöpp, *Helv. Chim. Acta*, 1931, **14**, 1431.
Heilbron *et al.*, *Biochem. J.*, 1932, **26**, 1178.
Hamano, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1937, **32**, 44.
Terada, *Chem. Abstracts*, 1934, **28**, 5182.
Heilbron, *J. Chem. Soc.*, 1948, 386.
Isler, *Chimia*, 1950, **4**, 103.
Attenburrow *et al.*, *J. Chem. Soc.*, 1952, 1094.

Vitamin A_2



$C_{20}H_{28}O$ MW, 284

Constituent of fresh-water fish-liver oils together with vitamin A_1. It has about 40% of the activity of cryst. vitamin A_1. Golden-yellow oil. Very labile to oxygen.
p-*Phenylazobenzoyl*: m.p. 78–90° (Kofler block); 84–7° (capillary).

Farrar, Hamlet, Henbest, Jones, *J. Chem. Soc.*, 1952, 503.
Shantz, Brinkman, *J. Biol. Chem.*, 1950, **183**, 467.
Morton, Salah, Stubbs, *Nature*, 1947, **159**, 744.

Vitamin A_1 Aldehyde.
See Retinene₁.

Vitamin A_2 Aldehyde.
See Retinene₂.

Vitamin B.
Numerous compounds have been reported in the literature, in addition to B_1 and B_2, to which has been assigned the generic term " Vitamin B ". Details of these compounds may be found in the following publications.

Robinson, *The Vitamin B Complex*, (John Wiley and Sons, Ltd., New York), 1951. (*Review.*)
Williams *et al.*, *The Biochemistry of B Vitamins*, (Reinhold Publishing Corporation, New York), 1951. (*Review.*)

Vitamin B_1.
See Aneurin.

Vitamin B_2.
See Lactoflavine.

Vitamin B_6.
See Adermin.

Vitamin B_{12} (*Cyanocobalamin*)

$C_{61-64}H_{86-92}O_{13}N_{14}PCo$ MW, about 1300

An anti-pernicious anæmia factor isolated from liver extracts and from the fermentation liquors of *Streptomyces griseus*. Red needles. $[\alpha]_{6563}^{23}$ $-59 \pm 9°$. The cobalt is trivalent and is co-ordinately linked. One co-ordinated group is the cyano group, which may be replaced by hydroxyl, nitrite, etc., giving rise to closely related compounds called hydroxocobalamin (vitamin B_{12a}, B_{12b}), nitrosocobalamin (vitamin B_{12c}), etc.

Brink *et al.*, *J. Am. Chem. Soc.*, 1949, **71**, 1854.
Smith, Parker, *Biochem. J.*, 1948, **43**, viii.
Snell, Wright, *Annual Reviews of Biochemistry*, 1950, **19**, 277 (*Review*).
Todd, *Chemistry and Industry*, 1951, 967.

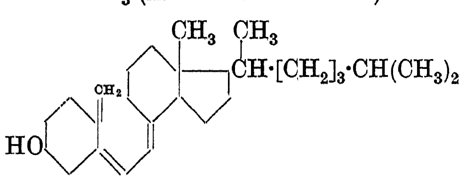
Vitamin B_c.
See Pteroylglutamic Acid.

Vitamin C.
See Ascorbic Acid.

Vitamin D_1.
Has been shown to be a mixture of Vitamin D_2 and lumisterol.

Vitamin D_2.
See Calciferol.

Vitamin D_3 (*Anti-rachitic vitamin*)



$C_{27}H_{44}O$ MW, 384

The vitamin D of fish-liver oils. Cryst. M.p. 87–8° (from Me_2CO); 63–4° (from MeOH). $[\alpha]_D^{20}$ $+83{\cdot}3°$ in Me_2CO. Shows absorption maximum at 265 mμ. As physiologically active as calciferol on rat test.

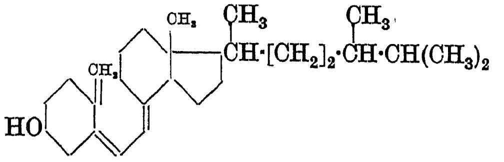
Acetyl: m.p. 131–2°. $[\alpha]_D^{18}$ $+142°$.

3 : 5-*Dinitrobenzoyl*: yellow needles. M.p. 131°. $[\alpha]_D^{20}$ $+20°$.

Allophanate: cryst. from Me_2CO. M.p. 173–4°.

Schenck, *Naturwissenschaften*, 1937, **10**, 159.
Windaus, Schenck, Werder, *Z. physiol. Chem.*, 1936, **241**, 100.

Vitamin D_4



$C_{28}H_{46}O$ MW, 398

Plates from Me_2CO.Aq. M.p. 107–8°. $[\alpha]_D^{18}$ $+89{\cdot}3°$ in Me_2CO. Shows absorption maximum at 2650 Å. Antirachitic activity about one-half that of calciferol on rat tests.

3 : 5-*Dinitrobenzoyl*: yellow needles. M.p. 135–6°. $[\alpha]_D^{18}$ $+94{\cdot}5°$ in Me_2CO.

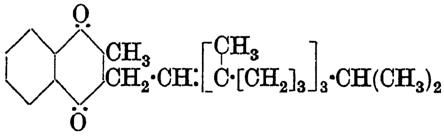
Windaus, Langer, *Ann.*, 1933, **508**, 105.
Windaus, Trautmann, *Z. physiol. Chem.*, 1937, **247**, 185.
Windaus, Güntzel, *Ann.*, 1939, **538**, 120.

Vitamin E.
See Tocopherol.

Vitamin H.
See Biotin.

Vitamin K.
See 2-Methyl-1 : 4-naphthoquinone.

Vitamin K_1 (2-*Methyl*-3-*phytyl*-1 : 4-*naphthoquinone*)



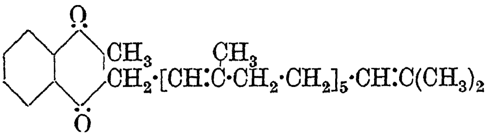
$C_{31}H_{46}O_2$ MW, 450

Fat soluble dietary factor essential for blood coagulation. Widely distributed in green leaves and vegetables, especially chestnut leaves and alfalfa. Lemon yellow oil which separates from Me_2CO or EtOH at $-70°$ in pale yellow rosettes, m.p. $-20°$. Sol. EtOH, Me_2CO, hexane, C_6H_6, $CHCl_3$. Spar. sol. MeOH. Insol. H_2O. NaOEt ⟶ blue col. Absorption maxima at 2400, 2480 (main), 2610, 2700 and 3230 Å. Reductive acetylation ⟶ diacetyl-dihydro-comp., m.p. 62–3°. Reductive benzoylation ⟶ dibenzoyl-dihydro-comp., m.p. 85–6°. CrO_3 ⟶ phthalic

acid + 2-methyl-1 : 4-naphthoquinone-3-acetic acid.

Doisy, Binkley, Thayer, *Chem. Reviews*, 1941, **28**, 477 (*Review and Bibl.*).
Fieser, *J. Am. Chem. Soc.*, 1939, **61**, 3467.

Vitamin K_2



Probable structure

$C_{41}H_{56}O_2$ MW, 580

Fat soluble dietary factor effective in controlling blood coagulation. Widely distributed in green leaves and vegetables. Isolated most readily from chestnut leaves and putrefied fish meal. Yellow cryst. from Me_2CO–EtOH or MeOH–$CHCl_3$. M.p. 51–2°. Slightly less sol. in org. solvents than vitamin K_1. Absorption spectrum similar to that of K_1. Reductive acetylation ⟶ diacetyl-dihydro-comp., m.p. 59·5–60°.

Binkley, McKee, Thayer, Doisy, *J. Biol. Chem.*, 1940, **133**, 721.
Doisy, Binkley, Thayer, *Chem. Reviews*, 1941, **28**, 477 (*Review and Bibl.*).

Vitamin K_6.
See under 2-Methyl-1 : 4-naphthylenediamine.

Vitamin M.
See Pteroylglutamic Acid.

Vitamin P (*Citrin*).
A substance affecting the permeability of capillary walls. Little is known about the chemical nature of the actual vitamin although several compounds, *e.g.* hesperidin, show some P activity.

Scarborough, *Biochem. J.*, 1945, **39**, 271.
Rosenberg, *The Chemistry and Physiology of the Vitamins*, (Interscience Publishers, New York).

Vitexin

$C_{15}H_{14}O_7$ MW, 306

Pigment from wood of *Vitex littoralis* or *Saponaria officinalis*. Yellow micro-plates from 40% Py. M.p. 263°.

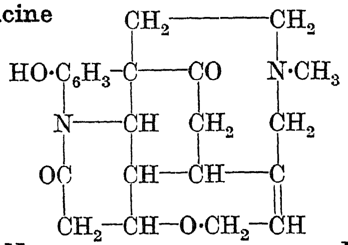
Acetyl deriv.: colourless prismatic needles from AcOH. M.p. 257–8°.

Perkin, *J. Chem. Soc.*, 1898, **73**, 1019.
Barger, *J. Chem. Soc.*, 1906, **89**, 1210.
Péteri, *J. Chem. Soc.*, 1939, 1635.

Volemitol.
See Sedoheptitol.

Voluntal.
See under Carbamic Acid.

Vomicine



$C_{22}H_{24}O_4N_2$ MW, 380

Strychnine alkaloid. Needles from EtOH.Aq. or EtOH–$CHCl_3$, prisms from Me_2CO. M.p. 278–80°. $[\alpha]_D^{22}$ + 80·4° in EtOH. Acid $FeCl_3$ ⟶ reddish-violet col. CrO_3 + H_2SO_4 ⟶ deep red sol. HNO_3 ⟶ brown col. Hot MeOH–KOH ⟶ green sol.

B,HCl: prisms from H_2O. M.p. 245° decomp.

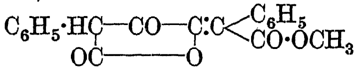
Methosulphate: cryst. from MeOH.Aq. M.p. 264° decomp.

Me ether: $C_{23}H_{26}O_4N_2$. MW, 394. Needles from EtOH. M.p. 286–90° decomp. $[\alpha]_D^{20}$ + 16·4° in EtOH.

Benzylidene deriv.: yellow plates from EtOH–$CHCl_3$. M.p. 280° decomp.

Wieland, Horner, *Ann.*, 1937, **528**, 73.
Wieland, Calvet, *Ann.*, 1931, **491**, 124.
Wieland, Oertel, *Ann.*, 1929, **469**, 193.
Bailey, Robinson, *Nature*, 1948, **161**, 433.
Huisgen, Wieland, Eder, *Ann.*, 1949, **561**, 193.

Vulpinic Acid (*Vulpic acid, pulvinic acid methyl ester*)



$C_{19}H_{14}O_5$ MW, 322

Occurs in many lichens. Exists in keto-form but can react as enol. Yellow plates from EtOH, needles or prisms from Et_2O. M.p. 148–9°. Very sol. $CHCl_3$. Mod. sol. Et_2O. Spar. sol. boiling EtOH. Insol. boiling H_2O. Hyd. ⟶ pulvinic acid. Reacts strongly acid and forms water stable salts. Poisonous.

Me ether: $C_{20}H_{16}O_5$. MW, 336. Needles from EtOH. M.p. 143–4°.

Et ether: $C_{21}H_{18}O_5$. MW, 350. Plates from EtOH. M.p. 138–9°.

Acetate: needles from EtOH or AcOH. M.p. 148° (156°).

Phenylacetate: needles from AcOH. M.p. 172°.

Benzoate: cryst. from AcOH. M.p. 176°.

Phenylurethane: yellow needles from EtOH. M.p. 237°.

Semicarbazone: yellow microcryst. M.p. 175°.

Asano, Kameda, *Ber.*, 1935, **68**, 1569.
Koller, Pfeiffer, *Monatsh.*, 1933, **62**, 164.
Karrer, Gehrckens, Heuss, *Helv. Chim. Acta*, 1926, **9**, 456.
Mazza, *Chem. Zentr.*, 1926, II, 1037.
Volhard, *Ann.*, 1894, **282**, 14.
Schenck, *ibid.*, 39.

W

Wilfordine

$C_{43}H_{49}O_{19}N$ MW, 883

Alkaloid occurring in *Tripterygium wilfordii*, Hook. Plates from Me_2CO–MeOH. M.p. 175–6°. $[\alpha]_D^{25}$ + 12° in Me_2CO.

Beroza, *J. Am. Chem. Soc.*, 1952, **74**, 3656; 1953, **75**, 44.

Wilforgine

$C_{41}H_{47}O_{19}N$ MW, 857

Alkaloid occurring in *Tripterygium wilfordii*, Hook. Plates from Me_2CO–MeOH. M.p. 211°. $[\alpha]_D^{25}$ + 25° in Me_2CO.

Beroza, *J. Am. Chem. Soc.*, 1952, **74**, 1585; 1953, **75**, 44.

Wilforine

$C_{43}H_{49}O_{18}N$ MW, 867

Alkaloid occurring in *Tripterygium wilfordii*, Hook. Plates from Me_2CO–MeOH. M.p. 169–70°. $[\alpha]_D^{25}$ + 30° in Me_2CO.

Beroza, *J. Am. Chem. Soc.*, 1952, **74**, 3656; 1953, **75**, 44.

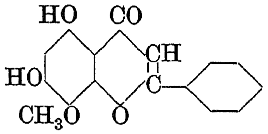
Wilfortrine

$C_{41}H_{47}O_{20}N$ MW, 873

Alkaloid occurring in *Tripterygium wilfordii*, Hook. Plates from Me_2CO–MeOH. M.p. 237·5—238°. $[\alpha]_D^{25}$ + 10° in Me_2CO.

Beroza, *J. Am. Chem. Soc.*, 1952, **74**, 1585; 1953, **75**, 44.

Wogonin (5 : 7-*Dihydroxy*-8-*methoxyflavone*)



$C_{16}H_{12}O_5$ MW, 284

Present in small amounts in roots of *Scutellaria baicalensis*, Georgi. Yellow needles from EtOH.Aq. or AcOEt–C_6H_6. M.p. 203°. Very sol. MeOH, EtOH, Me_2CO, AcOEt. Sol. Et_2O, AcOH, $CHCl_3$. Spar. sol. C_6H_6, H_2O. Insol. CS_2, ligroin.

Mono-Me ether: $C_{17}H_{14}O_5$. MW, 298. M.p. 183°.

Di-Me ether: $C_{18}H_{16}O_5$. MW, 312. Needles from H_2O. M.p. 167–8°.

Acetyl: m.p. 152–3°.

Benzoyl: m.p. 170°.

Hattori, Hayashi, *J. Chem. Soc. Japan*, 1933, **54**, 919.

Hattori, *Acta Phytochim.*, 1930, **5**, 99.

Rao, Rao, Seshadri, *Proc. Indian Acad. Sci.*, 1947, **26***A*, 13.

Wood sugar.

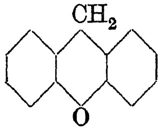
See Xylose.

X

Xanthaline.
See Papaveraldine.

Xanthanoic Acid.
See Xanthene-5-carboxylic Acid.

Xanthene (*Dibenzpyran, xanthane*)

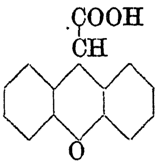


$C_{13}H_{10}O$ MW, 182

Leaflets from EtOH. M.p. 100·5° (98·5°). B.p. 310–12°. Sol. Et_2O, $CHCl_3$, C_6H_6, ligroin. Spar. sol. EtOH, AcOH, pet. ether. Insol. H_2O. Sublimes slowly below m.p. Volatile in steam. Ox. ⟶ xanthone.

Ipatiev, Orlov, Petrov, *Ber.*, 1927, **60**, 130.
Heller, v. Kostanecki, *Ber.*, 1908, **41**, 1325.

Xanthene-5-carboxylic Acid (*Xanthanoic acid*)



$C_{14}H_{10}O_3$ MW, 226

Needles from 50% EtOH. M.p. 223–4°.

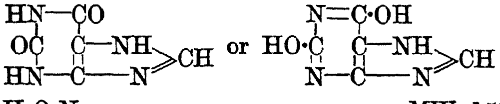
Me ester: $C_{15}H_{12}O_3$. MW, 240. Cryst. from MeOH. M.p. 85–6°.

Conant, Garvey, *J. Am. Chem. Soc.*, 1927, **49**, 2085.

Xanthenol-5.
See Xanthydrol.

Xanthic Acid.
See Xanthogenic Acid.

Xanthine (2 : 6-*Dihydroxypurine*)



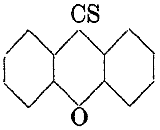
$C_5H_4O_2N_4$ MW, 152

Found in potatoes, coffee beans, etc. Cryst. powder. Sol. 1400 parts H_2O at 100°, 14,400 parts at 16°. Sol. 3000 parts EtOH at 17°. Very sol. KOH. Decomp. on heating. Very weak base. Conc. HCl at 220° ⟶ CO_2 + NH_3 + formic acid + glycine.

Perchlorate: cryst. + H_2O. Sinters at 255°, m.p. 262–4° decomp.

Frèrejacque, *Compt. rend.*, 1930, **191**, 950.
Biltz, Beck, *J. prakt. Chem.*, 1928, **118**, 166.
Baxter, Spring, *Nature*, 1944, **154**, 462.

Xanthione



$C_{13}H_8OS$ MW, 212

Red needles from C_6H_6. M.p. 156°. Sol. Et_2O, C_6H_6. Spar. sol. EtOH. Insol. H_2O. Conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Schönberg, Schütz, Nickel, *Ber.*, 1928, **61**, 1382.
Meyer, Szanecki, *Ber.*, 1900, **33**, 2580.

Xanthochelidonic Acid.
See 1 : 3 : 5-Triketopimelic Acid.

Xanthogenamide.
See under Thiourethane.

Xanthogenic Acid (*Dithiocarbonic O-ethyl ester, ethoxydithioformic acid, ethylxanthogenic acid, xanthic acid*)

$C_2H_5O{\cdot}CSSH$

$C_3H_6OS_2$ MW, 122

Unstable oily liq. F.p. about − 53°. Decomp. at 25° ⟶ CS_2 + C_2H_5OH.

NH_4 salt: unstable powder.

K salt: cryst. Sol. H_2O, EtOH. Prac. insol. Et_2O. D^{21} 1·558. Decomp. by boiling H_2O or by heat. Used as soil fumigant.

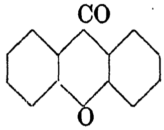
Me ester: $C_4H_8OS_2$. MW, 136. B.p. 182–3°. D_4^{25} 1·1189. Sol. EtOH, Et_2O. Insol. H_2O.

Et ester: $C_5H_{10}OS_2$. MW, 150. B.p. 199–200°, 76°/10 mm. Sol. EtOH, Et_2O. Insol. H_2O. D^{19} 1·085. n_D^{18} 1·5370.

Anhydride: golden-yellow needles from EtOH. M.p. 55°. Very sol. EtOH. Sol. Et_2O. Insol. H_2O.

Willcox, *J. Am. Chem. Soc.*, 1906, **28**, 1032.
Raag, *Chem.-Ztg.*, 1910, **34**, 83.
Reillen, Elben, Everet, *Ann.*, 1931, **485**, 50.
Great Western Electro-Chemical Co., U.S.P., 1,753,787, (*Chem. Abstracts*, 1930, **24**, 2471).

Xanthone (*Dibenz-γ-pyrone*)



$C_{13}H_8O_2$ MW, 196

Needles from EtOH. M.p. 174°. B.p. 349–50°/730 mm. Sol. $CHCl_3$, C_6H_6. Less sol. EtOH, Et_2O. Spar. sol. ligroin. Insol. cold

H_2O. Spar. volatile in steam. Conc. H_2SO_4 ⟶ yellow sol. with intense light blue fluor. Zn dust + NaOH ⟶ xanthydrol. Zn dust dist. ⟶ xanthene.

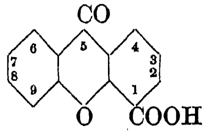
Oxime: cryst. M.p. 161°. Conc. H_2SO_4 ⟶ yellow sol. with blue fluor.

Phenylhydrazone: golden-yellow needles from EtOH. M.p. 152°. Conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Anil: golden-yellow cryst. from EtOH. M.p. 134·5°. Insol. alkalis. Conc. H_2SO_4 ⟶ yellow sol. with green fluor.

Perkin, *Ber.*, 1883, **16**, 339.
Graebe, *Ann.*, 1899, **254**, 280.
Graebe, Röder, *Ber.*, 1899, **32**, 1690.
Simon, *Bull. soc. chim. biol.*, 1926, **8**, 203.
Spektor, *Chem. Abstracts*, 1934, **28**, 575.
Holleman, *Organic Syntheses*, Collective Vol. I, 537.

Xanthone-1-carboxylic Acid



$C_{14}H_8O_4$ MW, 240

Needles from $PhNO_2$. M.p. 289° (275°). Sol. boiling AcOH, $PhNO_2$, anisole, phenetole. Mod. sol. boiling EtOH. Insol. $CHCl_3$, CCl_4, C_6H_6. Cold conc. H_2SO_4 ⟶ yellowish-green sol. Distils undecomp. Sublimes in needles. KOH fusion ⟶ salicylic acid.

Me ester: $C_{15}H_{10}O_4$. MW, 254. Yellowish needles from MeOH. M.p. 146·5°. Sol. MeOH. Insol. H_2O.

Et ester: $C_{16}H_{12}O_4$. MW, 268. Yellowish microneedles from EtOH. M.p. 123°.

Chloride: $C_{14}H_7O_3Cl$. MW, 258·5. Cryst. from *sym.*-tetrachloroethane. M.p. 165°.

Amide: $C_{14}H_9O_3N$. MW, 239. Powder. M.p. 320°.

Anilide: needles from EtOH. M.p. 252°.

Perkin, *J. Chem. Soc.*, 1883, **43**, 188.
Anschütz, Claasen, *Ber.*, 1922, **55**, 686.

Xanthone-3-carboxylic Acid.

Cryst. from AcOH or $PhNO_2$. M.p. 305°.

Me ester: needles from MeOH. M.p. 185°.

Et ester: needles from EtOH. M.p. 152°.

Chloride: pale yellowish needles from $CHCl_3$. M.p. 173°.

Amide: powder. M.p. 324° decomp.

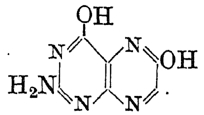
Anilide: needles from EtOH. M.p. 271°.

Anschütz, Stoltenhoff, Voeller, *Ber.*, 1925, **58**, 1740.

Xanthophyll.

This name is now employed as a generic term for hydroxylated carotenoids, *e.g.* lutein, zeazanthin, etc.

Xanthopterin (*Uropterin*



$C_6H_5O_2N_5$ MW, 179

Yellow wing pigment of butterflies, e.g. *Gonepteryx rhamni*, *Appias nero*, *Colias edusa*, etc. Amorph. hygroscopic mass. M.p. above 410° but carbonises above 360°. Prac. insol. H_2O. Readily sol. dil. NH_4OH and NaOH to yellow sols., and in 2*N*/HCl to colourless sol. H_2O_2 ⟶ leucopterin.

Wieland, Purrmann, *Ann.*, 1940, **544**, 163.
Schöpf, Kottler, *Ann.*, 1939, **539**, 128.
Schöpf, Becker, *Ann.*, 1936, **524**, 55, 126; 1933, **507**, 266.
Purrmann, *Ann.*, 1941, **548**, 284.
Elion, Light, Hitchings, *J. Am. Chem. Soc.*, 1949, **71**, 741.
Boon, Leigh, *J. Am. Chem. Soc.*, 1951, 1497.

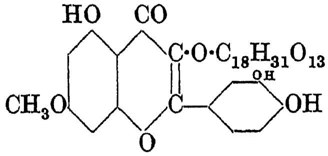
Xanthopurpurin.

See Purpuroxanthin.

Xanthoquininic Acid.

See 6-Hydroxycinchoninic Acid.

Xanthorhamnin (α-*Rhamnegin*)



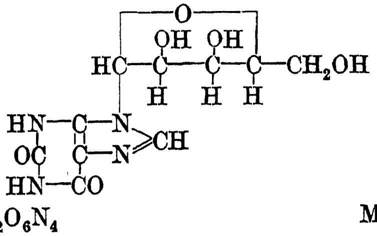
$C_{34}H_{42}O_{20}$ MW, 770

Yellow glycoside from Persian berries, *Rhamnus tinctoria*. Needles from EtOH–Et_2O. $[\alpha]_D$ + 3·75°. Sol. EtOH. Insol. Et_2O, $CHCl_3$, C_6H_6. Decomp. by H_2O. $FeCl_3$ ⟶ dark brown col. Hyd. ⟶ rhamnetin + rhamnose.

Tri-Me ether: $C_{37}H_{48}O_{20}$. MW, 812. Needles + $3H_2O$ from H_2O. M.p. 175–8°. Sol. H_2O. $FeCl_3$ ⟶ brown col.

Attree, Perkin, *J. Chem. Soc.*, 1927, 238.
Tanret, Tanret, *Bull. soc. chim.*, 1899, **21**, 1073.

Xanthosine (*Xanthine* 9-*ribofuranoside*)



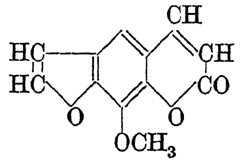
$C_{10}H_{12}O_6N_4$ MW, 284

A typical nucleoside. Prismatic rods + $2H_2O$ from H_2O. Decomp. on heating without melting. Sol. hot H_2O, EtOH.Aq. Spar. sol. cold H_2O. Hyd. by min. acids ⟶ xanthine + ribose.

Phosphoric ester: *see* Xanthylic Acid.

Levene, Jacobs, *Ber.*, 1910, **43**, 3163.
Gulland, Macrae, *J. Chem. Soc.*, 1933, 667.
Gulland, Holiday, Macrae, *J. Chem. Soc.*, 1934, 1640.

Xanthotoxin (*Zanthotoxin, ammoidin*)

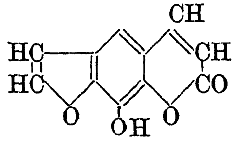


$C_{12}H_8O_4$ MW, 216

Constituent of fruit of *Fagara xanthoxyloides*, Linn. Prisms from 80% EtOH, needles from C_6H_6–pet. ether. M.p. 146° (144–5°). Very sol. boiling EtOH. Mod. sol. Me_2CO, AcOH. Spar. sol. H_2O, Et_2O, pet. ether. Spar. volatile in steam. Cold conc. H_2SO_4 ⟶ yellow sol. ⟶ brown on warming. Poisonous to fish.

Thoms, *Ber.*, 1911, **44**, 3325.
Späth, Pailer, *Ber.*, 1936, **69**, 767.

Xanthotoxol

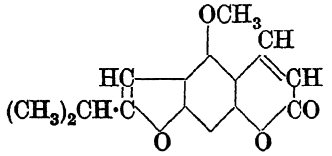


$C_{11}H_6O_4$ MW, 202

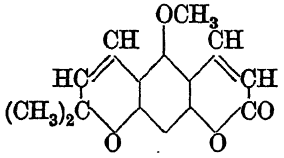
Present in seeds of *Angelica archangelica*. M.p. 251–2° (in evacuated tube). Diazomethane ⟶ xanthotoxin.

Späth, Vierhapper, *Ber.*, 1937, **70**, 248.

Xanthoxyletin (*Xanthoxylin-N*)



or



$C_{15}H_{14}O_4$ MW, 258

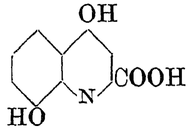
Constituent of bark of *Zanthoxylum americanum*, M. Prisms from pet. ether. M.p. 133°. Very sol. hot EtOH, $CHCl_3$, C_6H_6. Less sol. Me_2CO. Spar. sol. Et_2O. Sol. 49 parts cold EtOH, 25,000 parts cold H_2O. Cold conc. H_2SO_4 ⟶ red sol. Tasteless.

Dieterle, Kruta, *Chem. Zentr.*, 1937, I, 4243.
Gordin, *J. Am. Chem. Soc.*, 1906, **28**, 1650.
Bell, Robertson, Subramaniam, *J. Chem. Soc.*, 1936, 627.

Xanthoxylin-N.

See Xanthoxyletin.

Xanthurenic Acid (*Xanthuric acid*, 4 : 8-*dihydroxyquinoline*-2-*carboxylic acid*, 4 : 8-*dihydroxyquinaldinic acid*)



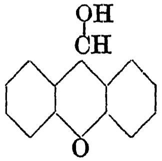
$C_{10}H_7O_4N$ MW, 205

Yellow micro-cryst. from H_2O. M.p. 286°. Sol. HCl, EtOH, AcOH + HCl. Spar. sol. usual solvents. Caustic alkalis and carbonates ⟶ yellow sols. Millon's reagent ⟶ red col. $FeSO_4$ ⟶ intense green col.

Me ester: $C_{11}H_9O_4N$. MW, 219. Yellow cryst. from MeOH. M.p. 262°. *Dibenzoyl*: m.p. 171°.

Musajo, *Chem. Zentr.*, 1935, II, 2079; 1936, II, 3540.
Musajo, Minchilli, *Ber.*, 1941, **74**, 1839.

Xanthydrol (*Dibenz-γ-pyranol*, 5-*hydroxyxanthene, xanthenol*-5)

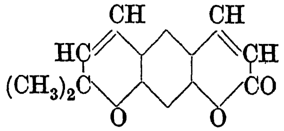


$C_{13}H_{10}O_2$ MW, 198

Needles from hot EtOH–H_2O. M.p. about 123°. Conc. H_2SO_4 ⟶ yellow sol. with green fluor. Warm in air ⟶ xanthone. Forms salts with min. acids. Reagent for urea.

Kirkhgof, Spektor, *Chem. Abstracts*, 1934, **28**, 5451.
Kyn-Jones, Ward, *J. Chem. Soc.*, 1930, 535.
Holleman, *Organic Syntheses*, Collective Vol. I, 539.
Lund, *Ber.*, 1937, **70**, 1520.

Xanthyletin

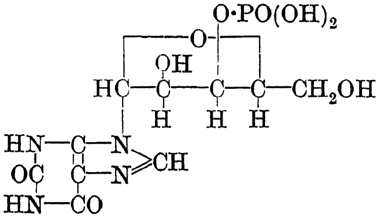


$C_{14}H_{12}O_3$ MW, 228

Constituent of bark of *Zanthoxylum americanum*, M. Prisms from petrol. M.p. 131·5°. B.p. 140–5°/0·1 mm. Conc. H_2SO_4 ⟶ orange-red sol. NaOH ⟶ acetone + resorcinol.

Bell, Robertson, *J. Chem. Soc.*, 1936, 1828.
Späth, Hillel, *Ber.*, 1939, **72**, 2093.

Xanthylic Acid (*Xanthine 9-ribosephosphoric acid*)



$C_{10}H_{15}O_9N_4P$ MW, 366

A typical nucleotide. Powder from H_2O. $[\alpha]_D^{20}$ − 61·66° in 5% NaOH. Aq. sol. at 50° decomp. with formation of *d*-ribose-phosphoric acid.

Brucine salt: needles from 30% EtOH. M.p. 200°.

Knopf, *Z. physiol. Chem.*, 1914, **92**, 160.
Levene, Dmochowski, *J. Biol. Chem.*, 1931, **93**, 565.
Levene, Harris, *J. Biol. Chem.*, 1932, **95**, 757.

Xaxaquin.
See under Quinine.

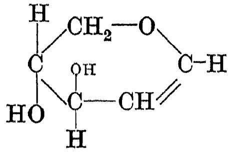
Xenylamine.
See Aminodiphenyl.

Xenylcarbinol.
See Hydroxymethyldiphenyl.

***p*-Xenyl Mercaptan.**
See 4-Mercaptodiphenyl.

Xeronic Acid.
See Diethylmaleic Acid.

Xylal (D-*Xylal*, D-*lyxal*)



$C_5H_8O_3$ MW, 116

Prisms from EtOH–Et_2O. M.p. 51° (49–50°). B.p. 109–12°/1·2 mm., 100–105° (bath temp.)/0·05 mm. $[\alpha]_D$ − 259° ± 3° in H_2O. Sol. H_2O, EtOH, Me_2CO. Spar. sol. Et_2O. Insol. C_6H_6.

Levene, Mori, *J. Biol. Chem.*, 1929, **83**, 803.
Overend, Shafizadeh, Stacey, *J. Chem. Soc.*, 1950, 1027.

Xylan

$(C_5H_8O_4)_n$ MW, $(132)_n$

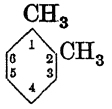
Occurs in wood gum, straw, esparto grass, maize cobs, oat hulls, hemp stalk, and is one of the hemicelluloses present in wood. The xylan from esparto grass is believed to consist of a singly branched molecule containing about 75 D-xylopyranose units joined by 1 : 4-β-linkages, the single branching point being formed by a 1 : 3-union. White powder. $[\alpha]_D^{21}$ − 92° in 2% NaOH.

Methylated deriv.: $[\alpha]_D^{20}$ − 85° in $CHCl_3$. Sol. AcOH, Me_2CO, $CHCl_3$. Spar. sol. Et_2O.

Acetylated deriv.: amorph. powder. $[\alpha]_D^{16}$ − 155° in *m*-cresol. Sol. Py. Spar. sol. $CHCl_3$. Insol. H_2O, MeOH, EtOH, Et_2O.

Chanda, Hirst, Jones, Percival, *J. Chem. Soc.*, 1950, 1289.

***o*-Xylene** (1 : 2-*Dimethylbenzene*)



C_8H_{10} MW, 106

Colourless mobile liq. M.p. − 25·0°. B.p. 143·95–144·15° (142·3°). Vap. press.: 4·5 mm. at 0°, 10 mm. at 20°, 34·5 mm. at 50°, 199·5 mm. at 100°. D_4^{20} 0·89679. $n_D^{16\cdot57}$ 1·50712. Sol. EtOH, Et_2O. $CrO_2Cl_2 \longrightarrow$ *o*-toluic aldehyde. $KMnO_4 \longrightarrow$ phthalic acid + *o*-toluic acid. CO + HCl in presence of $AlCl_3$ + $Cu_2Cl_2 \longrightarrow$ 3 : 4-dimethylbenzaldehyde.

Picrate: lemon-yellow cryst. from EtOH. M.p. 88·5°.

I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, **27**, 1366); F.P., 639,252, (*Chem. Abstracts*, 1929, **23**, 611).
General Aniline Works, U.S.P., 1,727,682, (*Chem. Abstracts*, 1929, **23**, 5196).

***m*-Xylene** (1 : 3-*Dimethylbenzene*).

Colourless, mobile liq. M.p. − 47·4°. B.p. 139·30°. Vap. press.: 2 mm. at 0°, 6 mm. at 20°, 31 mm. at 50°, 218 mm. at 100°. D_4^0 0·88113, D_4^{15} 0·86835. $n_{\text{He yellow}}^{15}$ 1·49989. Sol. EtOH, Et_2O. $CrO_3 \longrightarrow$ isophthalic acid.

Picrate: lemon-yellow cryst. from EtOH. M.p. 90–91·5°.

I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, **27**, 1366).

***p*-Xylene** (1 : 4-*Dimethylbenzene*).

Plates or prisms. M.p. 13–14°. B.p. 137·5–138·0°. Vap. press.: 2 mm. at 0°, 6 mm. at 20°, 32 mm. at 50°, 230 mm. at 100°. D_4^{28} 0·8541. n_D^{21} 1·5004. Sol. EtOH, Et_2O. Dil. $HNO_3 \longrightarrow$ *p*-toluic acid. $CrO_3 \longrightarrow$ terephthalic acid.

Picrate: lemon-yellow cryst. from EtOH. M.p. 90·5°.

Jannasch, *Ber.*, 1877, **10**, 1356.
I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, **27**, 1366); F.P., 639,252, (*Chem. Abstracts*, 1929, **23**, 611).
General Aniline Works, U.S.P., 1,727,682, (*Chem. Abstracts*, 1929, **23**, 5196).

Xyleneazocresol.
See 6-Hydroxy-3 : 2′ : 4′-trimethylazobenzene *and* 6-Hydroxy-3 : 3′ : 4′-trimethylazobenzene.

Xyleneazophenetole.
See under 4′-Hydroxy-2 : 4-dimethylazobenzene.

Xyleneazophenol.
See 4′-Hydroxy-2 : 4-dimethylazobenzene.

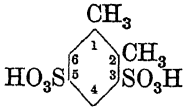
m-Xyleneazo-m-5-xylenol.
See 4-Hydroxy-2 : 6 : 2′ : 4′-tetramethylazobenzene.

Xyleneazoxylidine.
See Aminotetramethylazobenzene.

Xylene-carboxylic Acid.
See Dimethylbenzoic Acid.

Xylene-dicarboxylic Acid.
See Dimethylphthalic Acid, Dimethylisophthalic Acid, Dimethylterephthalic Acid *and* Phenylenediacetic Acid.

o-Xylene-3 : 5-disulphonic Acid (4 : 5-*Dimethylbenzene*-1 : 3-*disulphonic acid*)



$C_8H_{10}O_6S_2$ MW, 266

Dichloride: $C_8H_8O_4Cl_2S_2$. MW, 303. Yellow prisms from Et_2O. M.p. 79°. Sol. Et_2O, $CHCl_3$, CS_2.

Diamide: $C_8H_{12}O_4N_2S_2$. MW, 264. Cryst. M.p. 239°. Mod. sol. H_2O.

Dianilide: cryst. from EtOH. M.p. 199–200°. Very sol. Me_2CO. Sol. EtOH. Spar. sol. H_2O.

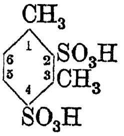
Pollak, Heimberg-Krauss, Katscher, Lustig, *Monatsh.*, 1930, **55**, 365.

o-Xylene-3 : 6-disulphonic Acid (2 : 3-*Dimethylbenzene*-1 : 4-*disulphonic acid*).

Diamide: cryst. from EtOH.Aq. M.p. 251°.

Holleman, Choufoer, *Chem. Abstracts*, 1924, **18**, 3183.

m-Xylene-2 : 4-disulphonic Acid (2 : 4-*Dimethylbenzene*-1 : 3-*disulphonic acid*)



$C_8H_{10}O_6S_2$ MW, 266

Dichloride: $C_8H_8O_4Cl_2S_2$. MW, 303. Viscous oil.

Diamide: $C_8H_{12}O_4N_2S_2$. MW, 264. Needles from H_2O. M.p. 223–4° (235°).

Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 247.

Choufoer, *Chem. Abstracts*, 1925, **19**, 2195.

m-Xylene-4 : 6-disulphonic Acid (4 : 6-*Dimethylbenzene*-1 : 3-*disulphonic acid*).

Needles. Decomp. very readily.

Di-Et ester: $C_{12}H_{18}O_6S_2$. MW, 322. Leaflets. Sol. EtOH. Insol. H_2O.

Difluoride: $C_8H_8O_4F_2S_2$. MW, 270. Cryst. M.p. 116–18°.

Dichloride: cryst. from pet. ether. M.p. 130°. Mod. sol. Et_2O, $CHCl_3$, CS_2.

Diamide: needles from H_2O. M.p. 249°. Spar. sol. H_2O. N : N′-*Di-Et*: needles from H_2O. M.p. 135°.

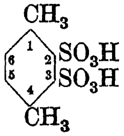
Dianilide: cryst. from 50% EtOH. M.p. 196°.

Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 244.

Davies, Dick, *J. Chem. Soc.*, 1931, 2107.

Holleman, Choufoer, *Rec. trav. chim.*, 1929, **48**, 1076.

p-Xylene-2 : 3-disulphonic Acid (3 : 6-*Dimethylbenzene*-1 : 2-*disulphonic acid*)



$C_8H_{10}O_6S_2$ MW, 266

Anhydride: $C_8H_8O_5S_2$. MW, 248. Leaflets from pet. ether. M.p. 189–90°.

Holleman, Choufoer, *Rec. trav. chim.*, 1929, **48**, 1082.

p-Xylene-2 : 5-disulphonic Acid (2 : 5-*Dimethylbenzene*-1 : 4-*disulphonic acid*).

Dichloride: cryst. from pet. ether. M.p. 164°.

Diamide: m.p. about 310° (297°).

Dianilide: cryst. from EtOH. M.p. 223°.

Pollak, Heimberg-Krauss, Katscher, Lustig, *Monatsh.*, 1930, **55**, 366.

p-Xylene-2 : 6-disulphonic Acid (2 : 5-*Dimethylbenzene*-1 : 3-*disulphonic acid*).

Needles. Very sol. H_2O.

Dichloride: $C_8H_8O_4Cl_2S_2$. MW, 303. Cryst. from ligroin. M.p. 81°. Sol. Et_2O, AcOEt, $CHCl_3$, C_6H_6. Spar. sol. CS_2, pet. ether, ligroin.

Diamide: $C_8H_{12}O_4N_2S_2$. MW, 264. Cryst. from EtOH.Aq. M.p. 294–5° decomp. Sol. Me_2CO. Spar. sol. Et_2O, AcOH, $CHCl_3$. Very spar. sol. H_2O.

Dianilide: cryst. from EtOH. M.p. 174°.

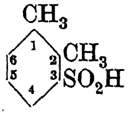
Holmes, *Am. Chem. J.*, 1891, **13**, 372.

Pollak, Schadler, *Monatsh.*, 1918, **39**, 144.

Pollak, Heimberg-Krauss, Katscher, Lustig, *Monatsh.*, 1930, **55**, 366.

Xylene Musk.
See 2 : 4 : 6-Trinitro-5-*tert.*-butyl-*m*-xylene.

o-Xylene-3-sulphinic Acid (2 : 3-*Dimethylbenzenesulphinic acid*)



$C_8H_{10}O_2S$ MW, 170

Cryst. M.p. 105°. Sol. Et_2O.

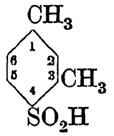
Moschner, *Ber.*, 1901, **34**, 1260.

o-Xylene-4-sulphinic Acid (3 : 4-*Dimethylbenzenesulphinic acid*).

Plates from H_2O. M.p. 83°.

Jacobsen, *Ber.*, 1877, **10**, 1010.
Knoevenagel, Kenner, *Ber.*, 1908, **41**, 3319.

m-Xylene-4-sulphinic Acid (2 : 4-*Dimethylbenzenesulphinic acid*)



$C_8H_{10}O_2S$ MW, 170

Needles from H_2O. M.p. 77–8°.

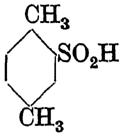
Gattermann, *Ber.*, 1899, **32**, 1141.
Knoevenagel, Kenner, *Ber.*, 1908, **41**, 3318.

m-Xylene-5-sulphinic Acid (3 : 5-*Dimethylbenzenesulphinic acid*).

Reddish cryst. mass. M.p. 75–6°.

Moschner, *Ber.*, 1901, **34**, 1260.

p-Xylenesulphinic Acid (2 : 5-*Dimethylbenzenesulphinic acid*)



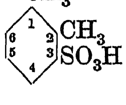
$C_8H_{10}O_2S$ MW, 170

Needles from H_2O. M.p. 85°. Sol. EtOH, Et_2O. Spar. sol. H_2O.

Anhydride: m.p. 68–9°.

Hilditch, *J. Chem. Soc.*, 1908, **93**, 1527.
Gattermann, *Ber.*, 1899, **32**, 1141.
Knoevenagel, Polack, *Ber.*, 1908, **41**, 3327.

o-Xylene-3-sulphonic Acid (2 : 3-*Dimethylbenzenesulphonic acid*)



$C_8H_{10}O_3S$ MW, 186

Cryst. from H_2O. Heat in air at 115–20° ⟶ 4-sulphonic acid.

Chloride: $C_8H_9O_2ClS$. MW, 204·5. Prisms from pet. ether. M.p. 47°.

Amide: $C_8H_{11}O_2NS$. MW, 185. Needles from H_2O. M.p. 167° (165°). Spar. sol. H_2O.

Moody, *Chem. News*, 1893, **67**, 34.
Moschner, *Ber.*, 1901, **34**, 1260.

o-Xylene-4-sulphonic Acid (3 : 4-*Dimethylbenzenesulphonic acid*).

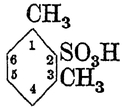
Plates or prisms + $2H_2O$ from $CHCl_3$. M.p. 63–4° (55°). Very hygroscopic.

Chloride: prisms from Et_2O. M.p. 51–2°.

Amide: prisms from EtOH. M.p. 143–4°.

Patterson, McMillan, Somerville, *J. Chem. Soc.*, 1924, **125**, 2489.
Jacobsen, *Ber.*, 1877, **10**, 1010.
Huntress, Autenrieth, *J. Am. Chem. Soc.*, 1941, **63**, 3446.

m-Xylene-2-sulphonic Acid (2 : 6-*Dimethylbenzenesulphonic acid*)



$C_8H_{10}O_3S$ MW, 186

Cryst. Heat at 100° ⟶ 4-sulphonic acid.

Chloride: $C_8H_9O_2ClS$. MW, 204·5. Prisms. M.p. 39°.

Amide: $C_8H_{11}O_2NS$. MW, 185. Needles. M.p. 113°.

Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 246.
Moody, *Chem. News*, 1888, **58**, 21.

m-Xylene-4-sulphonic Acid (2 : 4-*Dimethylbenzenesulphonic acid*).

Plates or prisms + $2H_2O$ from H_2O. M.p. 61–2°.

Fluoride: $C_8H_9O_2FS$. MW, 188. B.p. 246° (239–40°), 149–50°/15 mm. n_D^{20} 1·5086.

Chloride: cryst. M.p. 34°.

Amide: needles from H_2O. M.p. 138–9° (137°). N-*Benzoyl*: needles from EtOH. M.p. 149–51°.

Methylamide: $C_9H_{13}O_2NS$. MW, 199. Cryst. from EtOH. M.p. 43°. Sol. EtOH, Et_2O.

Dimethylamide: $C_{10}H_{15}O_2NS$. MW, 213. Cryst. from EtOH.Aq. M.p. 35°.

Anilide: cryst. from EtOH. M.p. 109–10°. N-*Me*: m.p. 55°.

p-*Nitroanilide*: cryst. + C_6H_6 from C_6H_6. M.p. 91–3°, solvent free 117–19°.

Anhydride: m.p. 139°. Decomp. on standing.

Crafts, *Ber.*, 1901, **34**, 1352.
Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 246.
Davies, Dick, *J. Chem. Soc.*, 1931, 2107.
Patterson, McMillan, Somerville, *J. Chem. Soc.*, 1924, **125**, 2489.
Huntress, Autenrieth, *J. Am. Chem. Soc.*, 1941, **63**, 3446.

m-Xylene-5-sulphonic Acid (3 : 5-*Dimethylbenzenesulphonic acid*).

Needles from H_2O.

Chloride: needles from pet. ether or C_6H_6. M.p. 94° (89–90°).

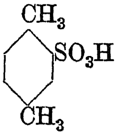
Bromide: $C_8H_9O_2BrS$. MW, 249. M.p. 92–3°.

Amide: prisms from boiling EtOH. M.p. 135°. Sol. EtOH. Less sol. Et_2O. Mod. sol. hot H_2O. Insol. ligroin.

Anilide: cryst. from EtOH. M.p. 119°.
p-*Toluidide*: prisms from EtOH. M.p. 121–2°.

Moschner, *Ber.*, 1901, **34**, 1260.
Jungahn, *Ber.*, 1902, **35**, 3756.
Armstrong, Wilson, *Chem. News*, 1901, **83**, 46.

p-Xylenesulphonic Acid (2 : 5-*Dimethylbenzenesulphonic acid*)

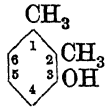


$C_8H_{10}O_3S$ MW, 186

Leaflets or prisms + $2H_2O$ from H_2O. M.p. 86°. Sol. $CHCl_3$.
Fluoride: $C_8H_9O_2FS$. MW, 188. M.p. 24·5°. B.p. 124–5°/21 mm.
Chloride: $C_8H_9O_2ClS$. MW, 204·5. Prisms. M.p. 24–6°.
Amide: $C_8H_{11}O_2NS$. MW, 185. Needles. M.p. 147–8°. Sol. EtOH. Mod. sol. hot H_2O.

Jacobsen, *Ber.*, 1877, **10**, 1009; 1878, **11**, 22.
Steinkopf *et al.*, *J. prakt. Chem.*, 1927, **117**, 39.
Huntress, Autenrieth, *J. Am. Chem. Soc.*, 1941, **63**, 3446.
Weber, *J. Chem. Education*, 1950, **27**, 384.

o-3-Xylenol (2 : 3-*Dimethylphenol*, 3-*hydroxy-o-xylene*)



$C_8H_{10}O$ MW, 122

Needles from EtOH.Aq. M.p. 75° (73·5°). B.p. 218°. $FeCl_3$ ⟶ blue col.
Me ether: 2 : 3-dimethylanisole. $C_9H_{12}O$. MW, 136. Cryst. M.p. 29°. B.p. 199°. Sol. EtOH, Et_2O, C_6H_6.
Et ether: 2 : 3-dimethylphenetole. $C_{10}H_{14}O$. MW, 150. M.p. 10°. B.p. 212·5°.
p-*Nitrobenzoyl*: m.p. 104·5°.

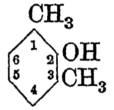
Moschner, *Ber.*, 1900, **33**, 742.
Short, Stromberg, Wiles, *J. Chem. Soc.*, 1936, 322.
Kruber, Schmitt, *Ber.*, 1931, **64**, 2270.

o-4-Xylenol (3 : 4-*Dimethylphenol*, 4-*hydroxy-o-xylene*).

Needles from H_2O. M.p. 62·5°. B.p. 225°/757 mm. Heat of comb. C_p 1035·4 Cal. $k = 5{\cdot}2 \times 10^{-11}$ at 25°.
Me ether: 3 : 4-dimethylanisole. B.p. 204–5°.
Et ether: 3 : 4-dimethylphenetole. B.p. 218°.
p-*Nitrobenzoyl*: m.p. 128°.
3 : 5-*Dinitrobenzoyl*: needles from 95% EtOH. M.p. 181·6°.
Phenylurethane: m.p. 119°.
1-*Naphthylurethane*: m.p. 141–2°.
Picrate: chrome yellow. M.p. 83·8°.

Jacobsen, *Ber.*, 1878, **11**, 28; 1884, **17**, 161.
Moschner, *Ber.*, 1900, **33**, 743.

m-2-Xylenol (2 : 6-*Dimethylphenol*, 2-*hydroxy-m-xylene*)



$C_8H_{10}O$ MW, 122

Leaflets or flat needles. M.p. 49°. B.p. 203°.
Me ether: 2 : 6-dimethylanisole. $C_9H_{12}O$. MW, 136. B.p. 182–3°.
Et ether: 2 : 6-dimethylphenetole. $C_{10}H_{14}O$. MW, 150. B.p. 195·5–196·5°. $D_4^{13\cdot9}$ 0·9420. $n_D^{13\cdot9}$ 1·497.
Propionyl: b.p. 112°/16 mm.
p-*Nitrobenzoyl*: m.p. 99°.
3 : 5-*Dinitrobenzoyl*: plates from 95% EtOH. M.p. 158·8°.
Picrate: orange-yellow. M.p. 50–3°.

Bamberger, *Ber.*, 1903, **36**, 2036.
Gattermann, *Ann.*, 1907, **357**, 363.
Carlin, Lauderl, *J. Am. Chem. Soc.*, 1950, **72**, 2762.

m-4-Xylenol (2 : 4-*Dimethylphenol*, 4-*hydroxy-m-xylene*).

Needles. M.p. 27–8°. B.p. 211·5°/766 mm., 97–8°/14 mm. D_4^{14} 1·0276. n_D^{14} 1·5420. Misc. with EtOH, Et_2O. Spar. sol. H_2O. Heat of comb. C_p 1037·5 Cal.
Me ether: 2 : 4-dimethylanisole. B.p. 192°. $D_4^{13\cdot45}$ 0·9691. $n_D^{12\cdot85}$ 1·517.
Et ether: 2 : 4-dimethylphenetole. B.p. 202–3°. $D_4^{13\cdot8}$ 0·9488. $n_D^{13\cdot95}$ 1·507.
Acetyl: b.p. 107·5–108·5°/13 mm. $D_4^{15\cdot5}$ 1·0298. $n_D^{15\cdot5}$ 1·4990.
Propionyl: b.p. 121·5°/15 mm. D^{17} 1·0104. n_D^{17} 1·4944.
Benzoyl: cryst. from AcOH. M.p. 37–8°. B.p. 110·5–111°/15 mm.
p-*Nitrobenzoyl*: m.p. 113°.
3 : 5-*Dinitrobenzoyl*: plates from 95% EtOH. M.p. 164·6°.
Phenylurethane: m.p. 102°.
1-*Naphthylurethane*: m.p. 134–5°.

Sabatier, Mailhe, *Compt. rend.*, 1910, **151**, 361.
Palfray, Duboc, *Compt. rend.*, 1927, **185**, 1479.

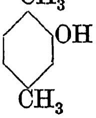
m-5-Xylenol (3 : 5-*Dimethylphenol*, 5-*hydroxy-m-xylene*).

Needles from H_2O. M.p. 68° (64°). B.p. 219·5°. Sublimes.
Me ether: 3 : 5-dimethylanisole. B.p. 193°.
Et ether: 3 : 5-dimethylphenetole. B.p. 208°.

Isopropyl ether: $C_{11}H_{16}O$. MW, 164. B.p. 208–10°.
Allyl ether: $C_{11}H_{14}O$. MW, 162. B.p. 109°/11 mm.
Acetyl: b.p. 130°/26 mm., 120°/11 mm.
Chloroacetyl: b.p. 175–7°/48 mm., 146–7°/12 mm.
3 : 5-*Dinitrobenzoyl*: rods from 95% EtOH. M.p. 195·4°.
Phenylurethane: m.p. 151°.

Raschig, D.R.P., 254,716, (*Chem. Zentr.*, 1913, I, 353).
Auwers, Borsche, *Ber.*, 1915, **48**, 1708, 1722.
Prelog, Metzler, Jeger, *Helv. Chim. Acta*, 1947, **30**, 675.

p-Xylenol (2 : 5-*Dimethylphenol*, *hydroxy*-p-*xylene*)



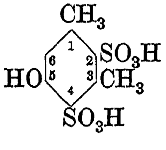
$C_8H_{10}O$ MW, 122

Prisms from EtOH–Et_2O. M.p. 75°. B.p. 211·5°/762 mm. Sol. EtOH, Et_2O. Heat of comb. C_p 1035·6 Cal. $k = 4{\cdot}8 \times 10^{-11}$ at 25°. No col. with $FeCl_3$. Sublimes. Volatile in steam.
Me ether: 2 : 5-dimethylanisole. $C_9H_{12}O$. MW, 136. B.p. 194°/772 mm.
Et ether: 2 : 5-dimethylphenetole. $C_{10}H_{14}O$. MW, 150. B.p. 198·8°/748 mm. Heat of comb. C_p 1368·85 Cal.
Acetyl: b.p. 237°/768 mm.
Propionyl: b.p. 125°/17 mm.
p-*Nitrobenzoyl*: m.p. 88°.
3 : 5-*Dinitrobenzoyl*: needles from 95% EtOH. M.p. 137·2°.
Phenylurethane: m.p. 162° (160–1°).
1-*Naphthylurethane*: m.p. 172–3°.
Picrate: orange. M.p. 81–2°.

Morgan, Pettet, B.P. 397,148, (*Chem. Abstracts*, 1934, **28**, 784).
Jacobsen, *Ber.*, 1878, **11**, 26.
Noelting, Witt, Forel, *Ber.*, 1885, **18**, 2665.

Xylenol-carboxylic Acid.
See Hydroxydimethylbenzoic Acid.

m-5-Xylenol-2 : 4-disulphonic Acid (3 : 5-*Dimethylphenol*-2 : 4-*disulphonic acid*, 6-*hydroxy*-2 : 4-*dimethylbenzene*-1 : 3-*disulphonic acid*)



$C_8H_{10}O_7S_2$ MW, 282

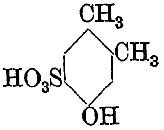
Dichloride: $C_8H_8O_5Cl_2S_2$. MW, 319. M.p. 117–19°.
Diamide: $C_8H_{12}O_5N_2S_2$. MW, 280. Leaflets from H_2O. M.p. 206–8°.
Dianilide: cryst. from C_6H_6. M.p. 205–7°.

Katscher, Lehr, *Monatsh.*, 1934, **64**, 238.
Ler, *Chem. Abstracts*, 1934, **28**, 4714.

m-5-Xylenol-4 : 6-disulphonic Acid (3 : 5-*Dimethylphenol*-2 : 6-*disulphonic acid*, 2-*hydroxy*-4 : 6-*dimethylbenzene*-1 : 3-*disulphonic acid*).
Dichloride: cryst. from pet. ether. M.p. 89–91°.
Dianilide: m.p. 160–1°.

Ler, *Chem. Abstracts*, 1934, **28**, 4714.

o-4-Xylenol-5-sulphonic Acid (3 : 4-*Dimethylphenol*-6-*sulphonic acid*, 2-*hydroxy*-4 : 5-*dimethylbenzenesulphonic acid*)

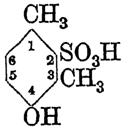


$C_8H_{10}O_4S$ MW, 202

Cryst. Steam at 107–10° ⟶ *o*-4-xylenol.
Na salt: prisms.
Ba salt: rhombic plates. Spar. sol. cold H_2O.

Jacobsen, *Ber.*, 1878, **11**, 28.

m-4-Xylenol-2-sulphonic Acid (2 : 4-*Dimethylphenol*-3-*sulphonic acid*, 3-*hydroxy*-2 : 6-*dimethylbenzenesulphonic acid*)



$C_8H_{10}O_4S$ MW, 202

Cryst.
Ba salt: needles.

Jacobsen, *Ann.*, 1879, **195**, 283.

m-4-Xylenol-5-sulphonic Acid (2 : 4-*Dimethylphenol*-6-*sulphonic acid*, 2-*hydroxy*-3 : 5-*dimethylbenzenesulphonic acid*).
Needles. Very sol. H_2O, EtOH. $FeCl_3$ ⟶ blue col. ⟶ green on adding EtOH. Decomp. with steam at 121–5°.
Fluoride: $C_8H_9O_3FS$. MW, 204. B.p. 71–3° in high vacuum.
Chloride: $C_8H_9O_3ClS$. MW, 220·5. *Acetyl*: cryst. from pet. ether. M.p. 62°.
Anilide: leaflets from EtOH.Aq. M.p. 142–3°. N-*Me*: prisms from pet. ether. M.p. 111–12°. N-*Acetyl*: cryst. from pet. ether. M.p. 105°

Katscher, Lehr, *Monatsh.*, 1934, **64**, 242.
Ler, *Chem. Abstracts*, 1934, **28**, 4715.

m-4-Xylenol-6-sulphonic Acid (2 : 4-*Dimethylphenol*-5-*sulphonic acid*, 3-*hydroxy*-4 : 6-*dimethylbenzenesulphonic acid*).
Needles. Very sol. H_2O, EtOH. $FeCl_3$ ⟶ bluish-violet col.

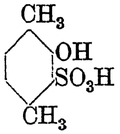
Me ether: $C_9H_{12}O_4S$. MW, 216. Needles. Sol. H_2O, EtOH, Et_2O. Spar. sol. C_6H_6. Decomp. on heating. *Amide*: $C_9H_{13}O_3NS$. MW, 215. Needles. M.p. 190°. Sol. EtOH. Spar. sol. H_2O.

Et ether: $C_{10}H_{14}O_4S$. MW, 230. Plates. Sol. EtOH. *Chloride*: $C_{10}H_{13}O_3ClS$. MW, 248·5. Rhombic plates from ligroin. M.p. 56°. *Amide*: $C_{10}H_{15}O_3NS$. MW, 229. Needles from EtOH. M.p. 169–70°.

Propyl ether: $C_{11}H_{16}O_4S$. MW, 244. Plates or needles. Mod. sol. H_2O, EtOH. *Amide*: $C_{11}H_{17}O_3NS$. Needles. M.p. 146°. Sol. EtOH. Spar. sol. H_2O.

Sartig, *Ann.*, 1885, **230**, 336.
Shober, Kiffer, *Am. Chem. J.*, 1897, **19**, 386.
Moody, *Chem. News*, 1892, **65**, 60.

p-2-Xylenol-3-sulphonic Acid (2 : 5-*Dimethylphenol*-6-*sulphonic acid*, 2-*hydroxy*-3 : 6-*dimethylbenzenesulphonic acid*)



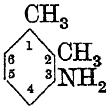
$C_8H_{10}O_4S$ MW, 202

Cryst. from H_2O. Decomp. with steam at 115–18°.

Ba salt: needles.

Morgan, Pettet, B.P. 397,148, (*Chem. Abstracts*, 1934, **28**, 784).
Jacobsen, *Ber.*, 1878, **11**, 27.

o-3-Xylidine (3-*Amino*-o-*xylene*)



$C_8H_{11}N$ MW, 121

Liq. B.p. 221–2°. D^{20} 0·9931. n_D^{20} 1·5684.

B,HCl: m.p. 254°.

N-*Me*: *see N*-Methyl-*o*-3-xylidine.

N-*Di-Me*: *see N*-Dimethyl-*o*-3-xylidine.

N-*Et*: $C_{10}H_{15}N$. MW, 149. B.p. 227–8°.

N-*Formyl*: m.p. 102°.

N-*Acetyl*: 3-acet-1 : 2-xylidide. Needles from EtOH or H_2O. M.p. 135° (131°). Sol. EtOH, Et_2O. Mod. sol. C_6H_6.

Hodgkinson, Limpach, *J. Chem. Soc.*, 1900, **77**, 68.
Menton, *Ann.*, 1891, **263**, 321.
Smith, Opie, *J. Org. Chem.*, 1941, **6**, 427.

o-4-Xylidine (4-*Amino*-o-*xylene*).

Plates or prisms from ligroin. M.p. 51° (47–8°). B.p. 226°. Mod. sol. pet. ether. Spar. sol. cold H_2O.

B,HCl: m.p. 256°.

N-*Formyl*: m.p. 52°.

N-*Di-Me*: *see N*-Dimethyl-*o*-4-xylidine.

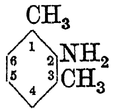
N-*Acetyl*: 4-acet-1 : 2-xylidide. Prisms or needles from EtOH.Aq. M.p. 99°. Very sol. EtOH.

N-*Chloroacetyl*: needles from EtOH.Aq. M.p. 109°. Sol. EtOH, Et_2O, AcOH.

N-*Cinnamoyl*: m.p. 175–6°.

Bamberger, Blangey, *Ann.*, 1911, **384**, 318, *Note* 2.
Jacobsen, *Ber.*, 1884, **17**, 160.
Graebe, *Ber.*, 1901, **34**, 1779.
Limpach, *Ber.*, 1888, **21**, 646.
Wisansky, Ansbacher, *Organic Syntheses*, 1948, XXVIII, 46.

m-2-Xylidine (2-*Amino*-m-*xylene*)



$C_8H_{11}N$ MW, 121

Liq. M.p. 11·2°. B.p. 214°/739 mm. D^{20} 0·9842. n_D^{20} 1·5610. Ox. ⟶ *m*-xyloquinone.

N-*Me*: *see N*-Methyl-*m*-2-xylidine.

N-*Di-Me*: *see N*-Dimethyl-*m*-2-xylidine.

N-*Et*: $C_{10}H_{15}N$. MW, 149. Oil. B.p. 217–18°.

N-*Di-Et*: $C_{12}H_{19}N$. MW, 177. B.p. 220–21°. Volatile in steam.

N-*Formyl*: needles from EtOH. M.p. 176–7° (rapid heat.). Mod. sol. EtOH.

N-*Acetyl*: 2-acet-1 : 3-xylidide. Needles. M.p. 177°. N-*Nitroso*: plates from pet. ether. M.p. 62–3°. Heat in C_6H_6 ⟶ 7-methylindazole.

N-*Benzoyl*: needles from EtOH.Aq. M.p. 168–168·5°. Very sol. EtOH, Et_2O, $CHCl_3$. Spar. sol. cold C_6H_6, ligroin.

Friedländer, Brand, *Monatsh.*, 1898, **19**, 639.
Busch, *Ber.*, 1899, **32**, 1008.
I.C.I., U.S.P., 1,867,962, (*Chem. Abstracts*, 1932, **26**, 5106); B.P. 328,418, (*Chem. Abstracts*, 1930, **24**, 5309).

m-4-Xylidine (4-*Amino*-m-*xylene*).

B.p. 215·8–216·0°/728 mm. $D_4^{19\cdot6}$ 0·9783. $n_D^{19\cdot6}$ 1·56066. Heat of comb. C_v 1111·42 Cal. $k = 6{\cdot}3 \times 10^{-10}$ at 15°.

B,HCl: prisms from H_2O. M.p. 235–6°.

Oxalate: m.p. 167°.

Picrate: yellow cryst. powder. M.p. 209° decomp.

N-*Me*: *see N*-Methyl-*m*-4-xylidine.

N-*Di-Me*: *see N*-Dimethyl-*m*-4-xylidine.

N-*Butyl*: $C_{12}H_{19}N$. MW, 177. Pale yellow oil. B.p. 267–70°/765 mm., 162–4°/29 mm. *Acetyl*: b.p. 290–3°/765 mm. *Benzoyl*: m.p. 193°.

N-*Formyl*: plates or needles from H_2O. M.p. 113–14°. Sol. EtOH, Et_2O.

N-*Acetyl*: 4-acet-1 : 3-xylidide. Needles from EtOH.Aq. M.p. 129–30°. B.p. 170°/10 mm. Sol. EtOH. Spar. sol. H_2O.

N-*Chloroacetyl*: needles from C_6H_6 or EtOH. M.p. 151–2°. Sol. EtOH. Spar. sol. C_6H_6.

N-*Benzoyl*: needles from EtOH. M.p. 192°. Sol. EtOH. Insol. H_2O.

N-p-*Nitrobenzoyl*: prisms from EtOH.Aq. M.p. 166°.

N-p-*Toluenesulphonyl*: m.p. 180·4–181·3°.

Pinnow, Oesterreich, *Ber.*, 1898, **31**, 2930.
Hodgkinson, Limpach, *J. Chem. Soc.*, 1900, **77**, 66.
Willgerodt, Schmierer, *Ber.*, 1905, **38**, 1473.
Reilly, O'Neill, *J. Soc. Chem. Ind.*, 1927, **46**, 226T.
Silesia Verein, F.P., 691,911, (*Chem. Abstracts*, 1931, **25**, 1265).

m-5-Xylidine (5-*Amino*-m-*xylene*).

M.p. 9·8°. B.p. 220–1°. D^{20} 0·9706. n_D^{20} 1·5581.

B,HCl: needles. Sublimes.

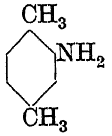
N-*Di-Me*: *see* *N*-Dimethyl-*m*-5-xylidine.

N-*Formyl*: prisms from EtOH.Aq. M.p. 76·5°.

N-*Acetyl*: 5-acet-1 : 3-xylidide. Needles from EtOH. M.p. 140·5° (144·5°).

Wroblewski, *Ann.*, 1881, **207**, 95.
Haller, Adams, Wherry, *J. Am. Chem. Soc.*, 1920, **42**, 184.

p-Xylidine (2-*Amino*-p-*xylene*)



$C_8H_{11}N$ MW, 121

Pale yellow leaflets. M.p. 15·5°. B.p. 213·5°. D_4^{21} 0·9790. $n_D^{21\cdot3}$ 1·55914. Mod. sol. H_2O. $k = 9{\cdot}63 \times 10^{-10}$ at 20°. Turns deep yellow in air. $CrO_3 \longrightarrow$ *p*-xyloquinone.

B,HCl: m.p. 228°.

Picrate: greenish-yellow. M.p. 171° decomp.

N-*Me*: *see* *N*-Methyl-*p*-xylidine.

N-*Di-Me*: *see* *N*-Dimethyl-*p*-xylidine.

N-*Et*: $C_{10}H_{15}N$. MW, 149. Oil. B.p. 222–3°/748 mm.

N-*Benzyl*: $C_{15}H_{17}N$. MW, 211. B.p. 320–5°.

N-*Formyl*: needles from H_2O. M.p. 116–17°.

N-*Acetyl*: 2-acet-1 : 4-xylidide. Needles from H_2O or toluene. M.p. 142°.

N-*Chloroacetyl*: needles from EtOH.Aq. M.p. 153°.

N-*Benzoyl*: needles from EtOH. M.p. 140°.

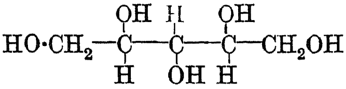
N-p-*Toluenesulphonyl*: m.p. 232–3°.

Pflug, *Ann.*, 1889, **255**, 172.
Noelting, Witt, Forel, *Ber.*, 1885, **18**, 2664.
Jannasch, *Ann.*, 1875, **176**, 55.
Schmidt, B.P. 252,460, (*Chem. Abstracts*, 1927, **21**, 2273).

β-Xylidinic Acid.

See 4-Methylisophthalic Acid.

Xylitol



$C_5H_{12}O_5$ MW, 152

Cryst. from EtOH in 2 forms. (i) Metastable rhombic cryst. M.p. 61–61·5°. (ii) Stable monoclinic cryst. M.p. 93–94·5°. Sweet taste. HI $\longrightarrow$ *sec.-n*-amyl iodide. PbO_2 + HCl $\longrightarrow$ xyloketose. Not oxidised by sorbose bacterium.

Di-isopropylidene deriv.: m.p. 34°. B.p. 80°/0·1 mm. n_D^{25} 1·4510.

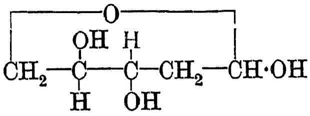
2 : 4-*Benzylidene deriv.*: m.p. 143–4°.

Dibenzylidene deriv.: cryst. M.p. 188°. Sol. $CHCl_3$. Prac. insol. H_2O, EtOH.

Penta-acetyl: m.p. 61·5–62·5°.

Fischer, *Ber.*, 1894, **27**, 2487.
Bertrand, *Bull. soc. chim.*, 1891, **5**, 555.
de Bruyn, v. Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151.
Bertrand, *Compt. rend.*, 1936, **203**, 143.
Wolfrom, Kohn, *J. Am. Chem. Soc.*, 1942, **64**, 1739.
Carson, Waisbrot, Jones, *J. Am. Chem. Soc.*, 1943, **65**, 1777.

2-Xylodesose (2-*Lyxodesose*)



$C_5H_{10}O_4$ MW, 134

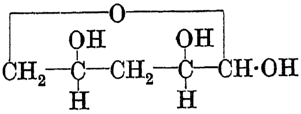
d-.

Plates. M.p. 92–6°. $[\alpha]_D^{22}$ − 40·25° $\longrightarrow$ + 50·75° in Py, − 22·5° $\longrightarrow$ − 2·0° in H_2O. Sol. H_2O, EtOH, Py. Spar. sol. Me_2CO. Insol. Et_2O, $CHCl_3$, CCl_4, C_6H_6. Reduces Fehling's.

Benzylphenylhydrazone: prisms or plates. M.p. 116–18°. $[\alpha]_D^{25}$ + 13·5° in Py. Sol. EtOH, Me_2CO, Py. Spar. sol. Et_2O. Insol. H_2O.

Levene, Mori, *J. Biol. Chem.*, 1929, **83**, 803.

3-Xylodesose (3-*Ribodesose*)



$C_5H_{10}O_4$ MW, 134

d-.

Syrup. $[\alpha]_D^{24}$ − 6·3° in H_2O. n_D^{16} 1·4610.

β-*Me-glycoside*: syrup. $[\alpha]_D^{19}$ − 13·3°. n_D^{26} 1·4566.

p-*Nitrophenylosazone*: red. cryst. from EtOH. M.p. 253–5°.

l-.

Syrup. $[\alpha]_D^{18} + 8{\cdot}7°$ in $CHCl_3$.

p-*Nitrophenylosazone*: dark red cryst. M.p. 254–6°.

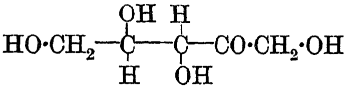
Kent, Stacey, Wiggins, *J. Chem. Soc.*, 1949, 1232.

Mukherjee, Todd, *J. Chem. Soc.*, 1947, 969.

p-Xylohydroquinone.

See 2 : 5-Dihydroxy-*p*-xylene.

Xyloketose



$C_5H_{10}O_5$ MW, 150

d-.

Syrup. Hygroscopic. $[\alpha]_D^{18} - 33{\cdot}2°$. Reduces Fehling's. p-*Bromophenylhydrazone*: m.p. 128–9°. $[\alpha]_D^{20} + 23{\cdot}7° \longrightarrow - 31{\cdot}2°$ in Py.

Isopropylidene deriv.: needles from Et_2O–pentane. M.p. 68°. B.p. 134°/0·5 mm.

l-.

Free sugar not isolated. Reduces Fehling's. $[\alpha]_D^{20} + 33{\cdot}1°$.

Phenylosazone: m.p. 160–3°. $[\alpha]_D + 0° 15'$ in Py–EtOH. Identical with *l*-xylosazone and *l*-lyxosazone.

p-*Bromophenylhydrazone*: yellow plates from dil. EtOH. M.p. 130–1° (128°). $[\alpha]^{20} - 25{\cdot}8° \longrightarrow + 31{\cdot}5°$ in Py. $[\alpha]_D - 1°$ in EtOH.

dl-.

Syrup. Sol. H_2O.

Phenylosazone: m.p. 210–15°. Identical with *dl*-xylosazone.

Methylphenylosazone: yellow needles. M.p. 173°.

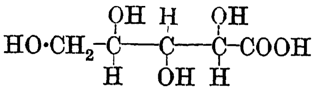
Neuberg, *Ber.*, 1902, **35**, 2628.

Levene, La Forge, *J. Biol. Chem.*, 1914, **18**, **319**.

v. Vargha, *Ber.*, 1935, **68**, 24.

Schmidt, Treiber, *Ber.*, 1933, **66**, 1765.

Xylonic Acid



$C_5H_{10}O_6$ MW, 166

d-.

Syrup. $[\alpha]_D + 17{\cdot}98°$ (final) in H_2O, $- 1{\cdot}9° \longrightarrow - 3{\cdot}6° \longrightarrow + 20{\cdot}2°$ in H_2O.

Cinchonine salt: plates from H_2O or needles from EtOH. M.p. 180° decomp. (170°). $[\alpha]_D^{17} + 125{\cdot}0°$ in H_2O.

Brucine salt: cryst. from EtOH. M.p. 176° (172–4°). $[\alpha]_D^{20} - 18{\cdot}7°$ in H_2O.

Morphine salt: needles from dil. EtOH. M.p. 153°.

Amide: $C_5H_{11}O_5N$. MW, 165. Plates. M.p. 81–2°. $[\alpha]_D^{16} + 44{\cdot}5° \longrightarrow + 23{\cdot}8°$ in H_2O. Sol. H_2O. Spar. sol. EtOH.

Phenylhydrazide: needles from AcOEt. M.p. 129° decomp.

γ-Lactone: $C_5H_8O_5$. MW, 148. Cryst. from Me_2CO. M.p. 99–103° (98–101°, 90–2°). $[\alpha]_D + 91{\cdot}8° \longrightarrow + 86{\cdot}7°$ in H_2O. *Triacetyl*: m.p. 99°. $[\alpha]_D^{16} + 62{\cdot}4°$ in EtOH.

Tetra-acetyl: m.p. 86–8°. $[\alpha]_D^{20} + 5°$ in EtOH, $- 2°$ in $CHCl_3$. *Nitrile*: laminæ from H_2O. M.p. 81·5°.

2 : 3-*Di-Me ether*: obtained only in sol. $[\alpha]_D^{22} + 30{\cdot}4° \longrightarrow + 63°$ in H_2O. *Phenylhydrazide*: needles. M.p. 107–8°. $[\alpha]_D^{23} + 30°$ in EtOH. p-*Bromophenylhydrazide*: needles. M.p. 150–1°. *γ-Lactone*: syrup. B.p. about 115°/0·02 mm. $[\alpha]_D^{23} + 97° \longrightarrow + 69°$ in H_2O. $n_D^{16\cdot5}$ 1·4640.

3 : 5-*Di-Me ether*: obtained only in sol. *Phenylhydrazide*: needles from C_6H_6. M.p. 94–5°. *γ-Lactone*: syrup. B.p. 105–6°/0·08 mm. $[\alpha]_{5780}^{21\cdot5} + 81{\cdot}5° \longrightarrow + 85{\cdot}1° \longrightarrow + 39°$ in H_2O. n_D^{15} 1·4643.

2 : 3 : 5-*Tri-Me ether*: obtained only in sol. $[\alpha]_{5780}^{17} + 42{\cdot}5° \longrightarrow + 40° \longrightarrow + 62{\cdot}5°$ in H_2O. *Phenylhydrazide*: needles from C_6H_6. M.p. 89–90°. *γ-Lactone*: syrup. B.p. 82°/0·06 mm. $[\alpha]_{5780}^{17} + 108° \longrightarrow + 110° \longrightarrow + 67{\cdot}5°$ in H_2O. n_D^{17} 1·4464.

2 : 3 : 4-*Tri-Me ether*: obtained only in sol. $[\alpha]_{5461}^{16} + 32{\cdot}7° \longrightarrow + 21{\cdot}5°$ in H_2O. *Phenylhydrazide*: m.p. 137–138·5°. *δ-Lactone*: needles from pet. ether. M.p. 56°. $[\alpha]_{5461}^{20}\ 0° \longrightarrow 21{\cdot}5°$ in H_2O, $[\alpha]_D^{15} - 3{\cdot}8° \longrightarrow + 20{\cdot}8°$ in H_2O.

l-.

Free acid not isolated.

Tetra-acetyl: m.p. 86–8°. $[\alpha]_D^{20} - 4{\cdot}5°$ in EtOH. *Nitrile*: cryst. from EtOH. M.p. 82°. Sol. EtOH, Et_2O, $CHCl_3$. Prac. insol. H_2O.

dl-.

Free acid not isolated.

Tetra-acetyl: m.p. 134–5°.

Allen, Tollens, *Ann.*, 1890, **260**, **306**.

Bertrand, *Bull. soc. chim.*, 1891, **5**, 556; 1896, **15**, 593.

Clowes, Tollens, *Ann.*, 1900, **310**, 175.

Maquenne, *Ann. chim. phys.*, 1901, **24**, 403.

Nef, Hedenburg, Glattfeld, *J. Am. Chem. Soc.*, 1917, **39**, 1650.

Haworth, Westgarth, *J. Chem. Soc.*, 1926, 880.

Haworth, Porter, *J. Chem. Soc.*, 1928, 616.

Hampton, Haworth, Hirst, *J. Chem. Soc.*, 1929, 1748.

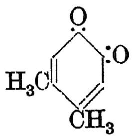
Deulofeu, *J. Chem. Soc.*, 1929, 2459.

Isbell, Frush, *Chem. Abstracts*, 1934, **28**, 1667.

Major, Cook, *J. Am. Chem. Soc.*, **1936**, **58**, 2476.

Xylopicric Acid.
See 2 : 4 : 6-Trinitro-*m*-5-xylenol.

***sym.-o*-Xylo-*o*-quinone** (4 : 5-*Dimethyl*-o-*benzoquinone*)

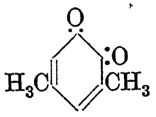


$C_8H_8O_2$ MW, 136

Exist in two forms. (i) Red cryst. from Et_2O. M.p. 102°. Spar. sol. C_6H_6, pet. ether. Sol. EtOH. (ii) Yellow plates.

Diepolder, *Ber.*, 1909, **42**, 2921.

***m*-Xylo-*o*-quinone** (3 : 5-*Dimethyl*-o-*benzoquinone*)

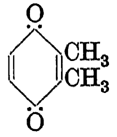


$C_8H_8O_2$ MW, 136

Dioxime: yellow needles from H_2O. M.p. 142° decomp. Sol. EtOH, AcOH, $CHCl_3$. Spar. sol. H_2O, petrol, C_6H_6.

Zincke, Schwarz, *Ann.*, 1899, **307**, 48.

***o*-Xylo-*p*-quinone** (2 : 3-*Dimethyl*-p-*benzoquinone*)



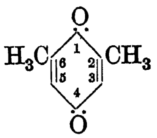
$C_8H_8O_2$ MW, 136

Yellow needles. M.p. 55°. Sublimes. Mod. sol. EtOH, Et_2O. Spar. sol. H_2O.

Monoxime: yellow needles. M.p. 166°. Very sol. MeOH, EtOH, C_6H_6, NH_3. Spar. sol. H_2O.

Noelting, Forel, *Ber.*, 1885, **18**, 2673.
Smith, Austin, *J. Am. Chem. Soc.*, 1942, **64**, 528.

***m*-Xylo-*p*-quinone** (2 : 6-*Dimethyl*-p-*benzoquinone*)



$C_8H_8O_2$ MW, 136

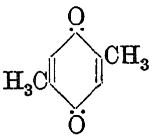
Yellow needles. M.p. 72–3°. Sublimes.

1-*Oxime*: yellow prisms from EtOH.Aq. M.p. 175°. Very sol. EtOH, Et_2O, C_6H_6, NH_3.

4-*Oxime*: yellow prisms from C_6H_6. M.p. 170–1°. Sol. EtOH, AcOH. Spar. sol. ligroin, C_6H_6. *Acetyl*: yellow cryst. from ligroin.

Noelting, Forel, *Ber.*, 1885, **18**, 2679.
Smith, Ungnade, *J. Org. Chem.*, 1939, **4**, 298.

***p*-Xylo-*p*-quinone** (2 : 5-*Dimethyl*-p-*benzoquinone, phlorone*)



$C_8H_8O_2$ MW, 136

Yellow needles from EtOH. M.p. 125°. Sol. Et_2O, $CHCl_3$, C_6H_6. Mod. sol. EtOH. Spar. sol. H_2O. Sublimes. Zn + AcOH ⟶ 2 : 5-dihydroxy-*p*-xylene.

Monoxime: yellow needles from H_2O. M.p. 173°. Sol. EtOH, Et_2O, C_6H_6. Very spar. sol. H_2O. *Me ether*: yellow cryst. M.p. 70·5–71°.

Dioxime: yellow needles from EtOH. M.p. about 272° (254°). Spar. sol. EtOH, AcOH, C_6H_6. Insol. H_2O. Sol. alkalis. *Diacetyl*: yellow prisms from AcOH. M.p. 170°.

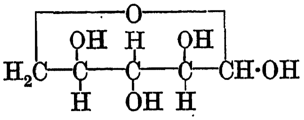
Monobenzoylphenylhydrazone: yellow prisms from ligroin. M.p. 122–4°; solidifies to orange cryst., m.p. 154–5°.

Veibel, Simesen, *Ber.*, 1930, **63**, 2480.
Auwers, Michaelis, *Ber.*, 1914, **47**, 1289.
Carstanjen, *J. prakt. Chem.*, 1881, **23**, 423.
Smith, Ungnade, *J. Org. Chem.*, 1939, **4**, 298.
Smith, Opie, *J. Org. Chem.*, 1941, **6**, 427.

***m*-Xylorcinol.**
See 4 : 6-Dihydroxy-*m*-xylene.

***p*-Xylorcinol.**
See 2 : 6-Dihydroxy-*p*-xylene.

Xylose (*Wood sugar*)



$C_5H_{10}O_5$ MW, 150

d-.

Needles. M.p. 144–5° (145–50°, 153–4°, 143°). Sweet taste. $[\alpha]_D^{20}$ + 92·0° ⟶ + 19·0° in H_2O. Sol. H_2O, hot EtOH. D^0 1·535. Heat of comb. C_v 3735 Cal. Reduces warm Fehling's. Does not ferment. Br water ⟶ *d*-xylonic acid. HNO_3 ⟶ xylotrihydroxyglutaric acid. Boil with dil. H_2SO_4 ⟶ furfural.

Semicarbazone: m.p. 202–4°. $[\alpha]_D^{20}$ − 38·8° ⟶ − 24·4° in H_2O. *Tetra-acetyl*: m.p. 232–3°. $[\alpha]_D^{20}$ + 21° in MeOH.

Phenylosazone: m.p. 159° (167° decomp., 164°, 161°). $[\alpha]_D$ − 40·9° in EtOH. Sol. Et_2O, Me_2CO. Spar. sol. H_2O. Identical with *d*-lyxosazone.

p-*Bromophenylosazone*: yellow needles. M.p. 208° (204°). Insol. Me_2CO.

o-*Tolylosazone*: m.p. 98–9°.

m-*Tolylosazone*: m.p. 137–8°.

p-*Tolylosazone*: m.p. 175–6°.

Phenylhydrazone: yellow cryst.

p-*Bromophenylhydrazone*: yellow cryst. M.p. 128°. $[\alpha]_D$ − 20·49° in H_2O.

2 : 4-*Dibromophenylhydrazone*: m.p. 127–8°.

p-*Nitrophenylhydrazone*: deep yellow cryst. M.p. 156° (154–5°). Sol. EtOH.

Methylphenylhydrazone: laminæ from AcOEt. M.p. 108–10° (103–5°). Sol. H_2O. Insol. C_6H_6.

Benzylphenylhydrazone: needles. M.p. 99° (95–100°). $[\alpha]_D$ − 33° in EtOH. Sol. EtOH, Et_2O. Spar. sol. H_2O.

Diphenylhydrazone: yellow plates from dil. EtOH or white needles from ligroin–Py. M.p. 128° (107–8°). Sol. hot H_2O, EtOH, Me_2CO, $CHCl_3$. Insol. Et_2O, ligroin.

2-*Naphthylhydrazone*: brown needles from MeOH. M.p. 123–4° (70°). $[\alpha]_D$ + 18·6° in MeOH, + 15·8° in AcOH. Sol. EtOH. Prac. insol. Et_2O, $CHCl_3$, C_6H_6.

α-*Methylglycoside*: α-methylxyloside. Cryst. M.p. 90–2°. $[\alpha]_D^{20}$ + 153·2° in H_2O (+ 153·9°). *Triacetyl*: m.p. 86°. $[\alpha]_D^{20}$ + 119·6° in $CHCl_3$. 2 : 3 : 4-*Tri-Me ether*: b.p. 110°/10 mm. n_D^{23} 1·4397. $[\alpha]_D^{20}$ + 121·5° in $CHCl_3$, + 112·7° in H_2O, + 122·2° in MeOH.

β-*Methylglycoside*: β-methylxyloside. Cryst. from AcOEt. M.p. 157° (155–6°). $[\alpha]_D^{20}$ − 65·9° in H_2O. *Triacetyl*: m.p. 115°. $[\alpha]_D^{20}$ − 60·8° in $CHCl_3$. 2 : 3 : 4-*Tri-Me ether*: m.p. 51°. B.p. 69–72°/0·5 mm. $[\alpha]_D^{20}$ − 69·5° in $CHCl_3$, − 81·7° in H_2O.

γ-*Methylglycoside*: γ-methylxyloside. Syrup. B.p. 161·5°/0·03 mm. $[\alpha]_D$ + 62·8° in EtOH.

2-*Me ether*: m.p. 134°. $[\alpha]_D^{14}$ + 34·5° in H_2O.

2 : 3-*Di-Me ether*: syrup. $[\alpha]_D^{20}$ + 22·6° ⟶ + 24° in H_2O. n_D^{20} 1·4783.

2 : 3 : 4-*Tri-Me ether*: prisms from AcOEt. M.p. 91–2°. $[\alpha]_D^{20}$ + 64·5° ⟶ + 17·7° in H_2O, + 55·8° ⟶ + 24·2° in $CHCl_3$, + 74° ⟶ + 21° in EtOH.

2 : 3 : 5-*Tri-Me ether*: liq. B.p. 110°/0·04 mm. $[\alpha]_D$ + 24·7° ⟶ + 31·2° in H_2O.

Triacetyl: cryst. from Et_2O. M.p. 138–41°. $[\alpha]_D^{23}$ + 70·11° ⟶ + 40·8° in $CHCl_3$.

Tetra-acetyl: β-*form*. M.p. 126–8°. $[\alpha]_D^{21}$ − 24·4° in $CHCl_3$.

Tribenzoyl: cryst. from C_6H_6. M.p. 188°. $[\alpha]_D^{20}$ + 39·5° in $CHCl_3$.

Tetrabenzoyl: α-form. Cryst. from C_6H_6. M.p. 115–16°. $[\alpha]_D^{20}$ + 115° in $CHCl_3$. β-*Form*. M.p. 178°. $[\alpha]_D^{22}$ − 47·45° in Me_2CO.

Acetone deriv.: m.p. 41–3°. $[\alpha]_D^{18}$ − 19·0°.

Diacetone deriv.: m.p. 44–5°. B.p. 85–7°/0·5 mm. $[\alpha]_D^{22}$ + 13·0° in H_2O, + 6·0° in $CHCl_3$.

l-.

Needles or prisms. M.p. 144° (141–3°). $[\alpha]_D^{20}$ − 79·3° ⟶ − 18·6° in H_2O.

Tetra-acetyl: α-*form*. M.p. 86°. $[\alpha]_D^{21}$ + 17·6° in $CHCl_3$. β-*Form*. M.p. 126°. $[\alpha]_D^{25}$ + 25·7° in $CHCl_3$. *Aldehydo form*. M.p. 90–1°. $[\alpha]_D^{20}$ + 22·5° in $CHCl_3$.

Tetrabenzoyl: α-*form*. M.p. 115–16°. $[\alpha]_D^{20}$ − 115° in $CHCl_3$. β-*Form*. M.p. 173–4°. $[\alpha]_D^{20}$ + 44·5° in $CHCl_3$.

Phenylosazone: m.p. 159–61°.

Mono-isopropylidene deriv.: b.p. 138°/13 mm.

Di-isopropylidene deriv.: cryst. from pentane. M.p. 43–5°. B.p. 135°/12 mm.

dl-.

Prisms. M.p. 129–31°.

Phenylosazone: yellow needles. M.p. 210–15° decomp. Identical with *dl*-lyxosazone.

Wheeler, Tollens, *Ann.*, 1889, **254**, 304.
Fischer, Stahel, *Ber.*, 1891, **24**, 528.
Schulze, Tollens, *Ann.*, 1892, **271**, 40.
Hudson, Harding, *J. Am. Chem. Soc.*, 1918, **40**, 1601.
Ling, Nanji, *J. Chem. Soc.*, 1923, **123**, 620.
Harding, *Sugar*, 1923, **25**, 124.
Carruthers, Hirst, *J. Chem. Soc.*, 1922, **121**, 2299.
Hirst, Purves, *J. Chem. Soc.*, 1923, **123**, 1352.
Haworth, Westgarth, *J. Chem. Soc.*, 1926, 880.
Hampton, Haworth, Hirst, *J. Chem. Soc.*, 1929, 1747.
Robertson, Speedie, *J. Chem. Soc.*, 1934, 824.
von Vargha, *Ber.*, 1935, **68**, 18.
Appel, *J. Chem. Soc.*, 1935, 425.
Sokolov, *Chem. Abstracts*, 1936, **30**, 5563.

6-αα-*d*-Xylosido-*d*-glucose.

See Isoprimeverose.

Xylosimine

$C_5H_{11}O_4N$ MW, 149

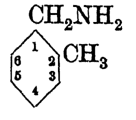
Cryst. M.p. 130° decomp. $[\alpha]_D$ − 18° 3′ ⟶ − 0° 46′ in H_2O.

de Bruyn, van Leent, *Rec. trav. chim.*, 1895, **14**, 144.
Levene, *J. Biol. Chem.*, 1916, **24**, 61.

Xylo-trihydroxyglutaric Acid.

See Trihydroxyglutaric Acid.

o-Xylylamine (o-*Methylbenzylamine*, ω-*amino*-o-*xylene*)



$C_8H_{11}N$ MW, 121

Oil. Cryst. in needles at − 20°. B.p. 205·5–206°/745 mm., 125°/105 mm. D_0^{19} 0·9768. n_D^{19} 1·5436.

B,HCl: plates from H_2O. M.p. 219–20°.

B,HBr: m.p. about 209°.

Sulphate: m.p. 176–9°.

B,HNO_3: m.p. 130°.

Oxalate: m.p. 94–5°.

$B,HAuCl_4$: m.p. about 180°.

B_2,H_2PtCl_6: yellow needles. M.p. 220–3° decomp.

N-*Acetyl*: needles from EtOH. M.p. 69°.
N-*Benzoyl*: needles from EtOH.Aq. M.p. 88°.
Picrate: yellow needles. M.p. 214–215·5°.

Konowalow, *J. Russ. Phys.-Chem. Soc.*, 1905, **37**, 536.
Strassmann, *Ber.*, 1888, **21**, 577.

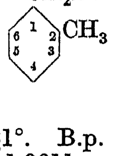
***m*-Xylylamine** (m-*Methylbenzylamine*, ω-*amino*-m-*xylene*).
Liq. B.p. 205–205·5°/750·5 mm., 96°/20 mm. Sol. EtOH, Et_2O. Insol. H_2O. D_0^{20} 0·9654.
B,HCl: needles from EtOH. M.p. 208°.
Sulphate: m.p. 248° decomp.
$B_2,(COOH)_2$: plates. M.p. 172°.
B_2,H_2PtCl_6: yellow plates. M.p. 214°.
N-*Acetyl*: liq. B.p. 235–40°.
N-*Benzoyl*: needles from $CHCl_3$–ligroin. M.p. 150–150·5° (70°).
Picrate: cryst. from MeOH. M.p. 198° decomp. (156°).

Shoppee, *J. Chem. Soc.*, 1932, 701.
Rupe, Bernstein, *Helv. Chim. Acta*, 1930, **13**, 462.
Sommer, *Ber.*, 1900, **33**, 1074.
Brömme, *Ber.*, 1888, **21**, 2701.

***p*-Xylylamine** (p-*Methylbenzylamine*, ω-*amino*-p-*xylene*).
M.p. 12·6–13·2°. B.p. 204°/739 mm. D_0^{20} 0·9520. n_D^{20} 1·53639. Spar. sol. H_2O.
B,HCl: needles. M.p. 234·5–235°.
Sulphate: plates from H_2O. M.p. about 130°.
$B,HAuCl_4$: yellow needles from H_2O. M.p. 169–71°.
B_2,H_2PtCl_6: plates from H_2O.
$B,HSnCl_3$: m.p. 107°.
N-*Acetyl*: cryst. from EtOH. M.p. 107–8°.
N-*Benzoyl*: needles from EtOH. M.p. 137°.
Picrate: cryst. from H_2O. M.p. 204° decomp.

Druce, *J. Chem. Soc.*, 1918, **113**, 718.
Curtius, Darapsky, *Ber.*, 1902, **35**, 3232.
Lustig, *Ber.*, 1895, **28**, 2988.
Métayer, *Ann. chim.*, 1949, **4**, 196.

***o*-Xylyl bromide** (o-*Methylbenzyl bromide*, ω-*bromo*-o-*xylene*)



C_8H_9Br MW, 185

Prisms. M.p. 21°. B.p. 216–17°/742 mm., 108°/16 mm. D^{23} 1·3811.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, **91**, 1695.

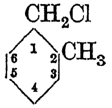
***m*-Xylyl bromide** (m-*Methylbenzyl bromide*, ω-*bromo*-m-*xylene*).
Liq. B.p. 212·5° (slight decomp.), 185°/340 mm., 100–1°/14 mm. D^{23} 1·3711.

Titley, *J. Chem. Soc.*, 1926, 514.
Haller, Bauer, *Compt. rend.*, 1911, **153**, 22.
Atkinson, Thorpe, *J. Chem. Soc.*, 1907, **91**, 1696.

***p*-Xylyl bromide** (p-*Methylbenzyl bromide*, ω-*bromo*-p-*xylene*).
Needles from EtOH. M.p. 35°. B.p. 218–20°/740 mm. Sol. Et_2O, $CHCl_3$.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, **91**, 1697.

***o*-Xylyl chloride** (o-*Methylbenzyl chloride*, ω-*chloro*-o-*xylene*)



C_8H_9Cl MW, 140·5
Liq. B.p. 197–9° (195–203°).

Reyman, *Bull. soc. chim.*, 1876, **26**, 534.

***m*-Xylyl chloride** (m-*Methylbenzyl chloride*, ω-*chloro*-m-*xylene*).
Liq. B.p. 195–6°. D^{20} 1·064. n_D^{25} 1·5327.

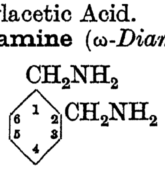
King, Merriam, *Chem. Zentr.*, 1935, II, 2359.
Gundelach, *Bull. soc. chim.*, 1876, **26**, 43.
Asolkar, Guha, *J. Indian Chem. Soc.*, 1946, **23**, 47.

***p*-Xylyl chloride** (p-*Methylbenzyl chloride*, ω-*chloro*-p-*xylene*).
Fuming liq. B.p. 200–2°, 90°/20 mm.

Curtius, Sprenger, *J. prakt. Chem.*, 1900, **62**, 111.
Shorygin, Bogdanova, *Chem. Abstracts*, 1939, **33**, 4216.

Xylyl cyanide.
See under Tolylacetic Acid.

***o*-Xylylenediamine** (ω-*Diamino*-o-*xylene*)



$C_8H_{12}N_2$ MW, 136
Liq. with ammoniacal odour. Absorbs CO_2 from the air.
N-*Tetra-Me*: $C_{12}H_{20}N_2$. MW, 192. Liq. B.p. 105–6°/14 mm. *Dimethiodide*: m.p. 219° decomp. *Dimethobromide*: prisms from EtOH–Et_2O. M.p. 207–8°. *Picrate*: yellow cryst. M.p. 187–8°.
N-*Tetra-Et*: $C_{16}H_{28}N_2$. MW, 248. Liq. B.p. 170–5°/20 mm.

N : N′-*Diphenyl* : $C_{20}H_{20}N_2$. MW, 288. Plates from EtOH. M.p. 114°. Sol. EtOH, C_6H_6. Insol. H_2O, ligroin. N : N′-*Di-Me* : $C_{22}H_{24}N_2$. MW, 316. Plates from EtOH. M.p. 110°.

N-*Tetraphenyl* : $C_{32}H_{28}N_2$. MW, 440. Needles from Me_2CO.Aq. M.p. 179°. Sol. Me_2CO, $CHCl_3$. Spar. sol. EtOH.

N : N′-*Diacetyl* : cryst. from Et_2O. M.p. 146°.

N : N′-*Dibenzoyl* : needles from EtOH. M.p. 184°.

Picrate : yellow needles. Decomp. at 170°.

Gabriel, Pinkus, *Ber.*, 1893, **26**, 2212.
Strassmann, *Ber.*, 1888, **21**, 579.

***m*-Xylylenediamine** (ω-*Diamino*-m-*xylene*).
Liq. B.p. 245–8°, 140°/14 mm. Misc. with EtOH, Et_2O.

B,2HBr : yellowish needles. M.p. 266°.

N : N′-*Diacetyl* : cryst. from C_6H_6. M.p. 134–5°.

N : N′-*Dibenzoyl* : m.p. 172°.

Picrate : yellow plates. Decomp. at 185–90°.

Brömme, *Ber.*, 1888, **21**, 2705.
Ruggli, Leupin, Dahn, *Helv. Chim. Acta*, 1947, **30**, 1845.

***p*-Xylylenediamine** (ω-*Diamino*-p-*xylene*).
Cryst. M.p. 35°.

B,H₂PtCl₆ : cryst. Decomp. at 250°.

N-*Tetraphenyl* : $C_{32}H_{28}N_2$. MW, 440. Needles from AcOH. M.p. 186°. Sol. AcOH. Mod. sol. Me_2CO. Spar. sol. EtOH.

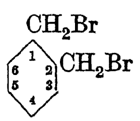
N-*Tetra-acetyl* : needles from EtOH. M.p. 194°.

N : N′-*Dibenzoyl* : needles. M.p. 193–4°.

Picrate : orange needles. Decomp. at 232°.

Lustig, *Ber.*, 1895, **28**, 2992.

***o*-Xylylene dibromide** (o-*Xylylene bromide*, ω-*dibromo*-o-*xylene*)



$C_8H_8Br_2$ MW, 264

Cryst. from $CHCl_3$. M.p. 95°. Decomp. on dist. Sol. EtOH, Et_2O, $CHCl_3$. Mod. sol. pet. ether.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, **91**, 1696.

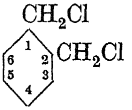
***m*-Xylylene dibromide** (m-*Xylylene bromide*, ω-*dibromo*-m-*xylene*).
Prisms from Me_2CO. M.p. 77°. B.p. 158–60°/12 mm. Sol. Et_2O, $CHCl_3$, ligroin. D^0 1·959.

Titley, *J. Chem. Soc.*, 1926, 514.
Braun, Karpf, Garn, *Ber.*, 1920, **53**, 101.

***p*-Xylylene dibromide** (p-*Xylylene bromide*, ω-*dibromo*-p-*xylene*).
Cryst. from $CHCl_3$ or C_6H_6. M.p. 145–7°. B.p. 245°. D^0 2·012. Sol. $CHCl_3$. Spar. sol. Et_2O.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, **91**, 1698.
Goodson, *Brit. J. Pharmacol.*, 1948, **3**, 62.
Ruggli, Theilheimer, *Helv. Chim. Acta*, 1941, **24**, 899.

***o*-Xylylene dichloride** (o-*Xylylene chloride*, ω-*dichloro*-o-*xylene*)



$C_8H_8Cl_2$ MW, 175

Cryst. from pet. ether. M.p. 55°. B.p. 239–41°, 130–5°/19 mm. Sol. EtOH, Et_2O, $CHCl_3$, ligroin. D^0 1·393.

Quelet, *Bull. soc. chim.*, 1933, **53**, 222.

***m*-Xylylene dichloride** (m-*Xylylene chloride*, ω-*dichloro*-m-*xylene*).
Cryst. M.p. 34·2°. B.p. 250–5°. D^{20} 1·302.

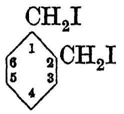
Colson, Gautier, *Ann. chim.*, 1887, **11**, 23.

***p*-Xylylene dichloride** (p-*Xylylene chloride*, ω-*dichloro*-p-*xylene*).
Plates from EtOH. M.p. 100°. B.p. 240–5° decomp., 135°/16 mm. D^0 1·417.

Quelet, *Bull. soc. chim.*, 1933, **53**, 222.
Sabetay, *Compt. rend.*, 1931, **192**, 1109.
Grimaux, *Ann.*, 1870, **155**, 340.
Kulka, *Chem. Abstracts*, 1945, **39**, 4319.

Xylylene dicyanide.
See under Phenylenediacetic Acid.

***o*-Xylylene di-iodide** (o-*Xylylene iodide*, ω-*di-iodo*-o-*xylene*)



$C_8H_8I_2$ MW, 358

Yellow prisms from Et_2O. M.p. 109–10°.

Finkelstein, *Ber.*, 1910, **43**, 1532.

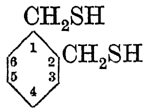
***m*-Xylylene di-iodide** (m-*Xylylene iodide*, ω-*di-iodo*-m-*xylene*).
M.p. 106°.

Finkelstein, *Ber.*, 1910, **43**, 1532.

***p*-Xylylene di-iodide** (p-*Xylylene iodide*, ω-*di-iodo*-p-*xylene*).
M.p. 175° decomp. Sol. hot EtOH, $CHCl_3$. Spar. sol. Et_2O.

Knoll, D.R.P., 230,172, (*Chem. Zentr.*, 1911, I, 359).
Finkelstein, *Ber.*, 1910, **43**, 1532.

o-Xylylene Dimercaptan (*Dithio*-o-*xylylene glycol*, ω-*dimercapto*-o-*xylene*)



$C_8H_{10}S_2$ MW, 170

Prisms. M.p. 45–6°. B.p. 160°/20 mm. Sol. EtOH, Et_2O. Spar. sol. pet. ether.

Autenrieth, Hennings, *Ber.*, 1901, **34**, 1774.

m-Xylylene Dimercaptan (*Dithio*-m-*xylylene glycol*, ω-*dimercapto*-m-*xylene*).

Liq. with characteristic odour. B.p. 157–8°/15 mm.

Dibenzoyl: prisms. M.p. 52·5°.

Autenrieth, Beuttel, *Ber.*, 1909, **42**, 4358.

p-Xylylene Dimercaptan (*Dithio*-p-*xylylene glycol*, ω-*dimercapto*-p-*xylene*).

Cryst. M.p. 46–7°. B.p. 156°/12 mm.

Dibenzoyl: needles. M.p. 135°.

Autenrieth, Beuttel, *Ber.*, 1909, **42**, 4349.

o-Xylylene oxide.

See Phthalan.

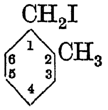
Xylylic Acid.

See Dimethylbenzoic Acid.

Xylylidene chloride.

See Methylbenzylidene chloride.

o-Xylyl iodide (o-*Methylbenzyl iodide*, ω-*iodo*-o-*xylene*)



C_8H_9I MW, 232

Needles from Et_2O. M.p. 34°. Decomp. on dist.

Zeltner, Tarassow, *Ber.*, 1910, **43**, 945.

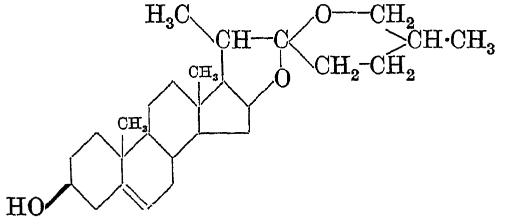
p-Xylyl iodide (p-*Methylbenzyl iodide*, ω-*iodo*-p-*xylene*).

Needles from Et_2O. M.p. 45·5–46·5°.

Zeltner, Tarassow, *Ber.*, 1910, **43**, 944.

Y

Yamogenin



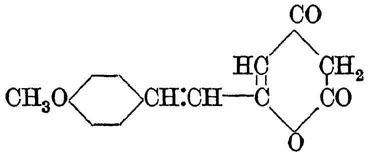
$C_{27}H_{42}O_3$ MW, 414

Sapogenin isolated from the rhizomes of Mexican *dioscorea.* M.p. 200° (201°).

Acetyl: m.p. 182°.

Marker *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2184.

Yangonalactone



$C_{14}H_{12}O_4$ MW, 244

Yellow prisms from MeOH or EtOH. M.p. 238°. Sol. Et_2O, AcOEt, AcOH, C_6H_6. Forms derivs. of enolic form.

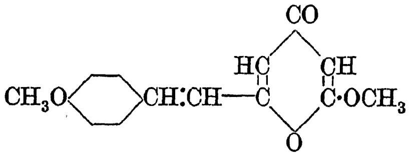
Me ether: *see* Yangonin.

O-*Acetyl*: red prisms from AcOEt. M.p. 133°.

O-*Benzoyl*: yellow leaflets from EtOH. M.p. 147°.

Borsche, Blount, *Ber.*, 1932, **65**, 820.
Borsche, Bodenstein, *Ber.*, 1929, **62**, 2519.

Yangonin

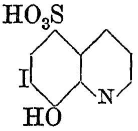


$C_{15}H_{14}O_4$ MW, 258

Constituent of Kawa root. Greenish-yellow cryst. with blue fluor. from MeOH or Me_2CO. M.p. 153–4°. Sol. Me_2CO, AcOH. Spar. sol. Et_2O, CS_2. Sol. conc. $H_2SO_4 \longrightarrow$ yellow sol. with green fluor.

Jablonski, *Chem. Abstracts*, 1935, **29**, 7982.
Borsche, Bodenstein, *Ber.*, 1929, **62**, 2519.
Murakami, *Chem. Abstracts*, 1918, **12**, 2547.

Yatren (*Loretin, quinoxyl,* 7-*iodo*-8-*hydroxyquinoline*-5-*sulphonic acid*)

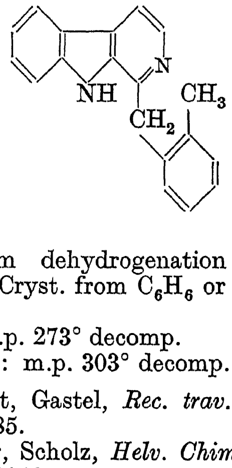


$C_9H_6O_4NIS$ MW, 351

Yellow prisms or laminæ. Decomp. at about 260°. Very spar. sol. H_2O, EtOH. Prac. insol. Et_2O, $CHCl_3$, C_6H_6. Sol. conc. H_2SO_4. HNO_3 (D 1·52) $\longrightarrow$ 5 : 7-dinitro-8-hydroxyquinoline. Forms cryst. salts with Na, K, Mg, Ca, Ba, Sr. Used as disinfectant in treatment of amœbic dysentery, etc.

I.G., D.R.P., 545,915, (*Chem. Abstracts*, 1933, **27**, 566).
Claus, *Arch. Pharm.*, 1893, **231**, 706.

Yobyrine (2-o-*Methylbenzyl*-β-*carboline*)



$C_{19}H_{16}N_2$ MW, 272

A selenium dehydrogenation product of yohimbine. Cryst. from C_6H_6 or EtOH. M.p. 218–19°.

B,HCl: m.p. 273° decomp.

Methiodide: m.p. 303° decomp.

Wibaut, Gastel, *Rec. trav. chim.*, 1935, **54**, 85.
Barger, Scholz, *Helv. Chim. Acta*, 1933, **16**, 1346.
Clemo, Swan, *J. Chem. Soc.*, 1946, 617.

Yohimbene

$C_{21}H_{26}O_3N_2$ MW, 354

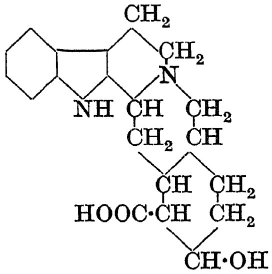
Isomeric with yohimbine. Leaflets from MeOH. M.p. 278° decomp. Sol. MeOH, EtOH. Spar. sol. Me_2CO, $CHCl_3$, Et_2O. Insol. AcOEt, petrol, C_6H_6, toluene. [α] + 43·7° in Py. Hyd. with loss of $CO_2 \longrightarrow$ yohimbol. Extremely poisonous.

B,HCl: needles + $3H_2O$ from MeOH.Aq. M.p. 234°. [α] − 8·8° in H_2O.

B,MeI: needles + $4H_2O$ from Et_2O–EtOH. Decomp. at 288°.

Hahn, Stenner, *Ber.*, 1928, **61**, 282.
Hahn, Brandenberg, *Ber.*, 1926, **59**, 2189.

Yohimbic Acid



$C_{20}H_{24}O_3N_2$ MW, 340

Cryst. + $1H_2O$ from H_2O. M.p. 256° (265–9°). $[\alpha]_D^{20}$ + 138·8° in Py. Loss of CO_2 ⟶ yohimbol.

Me ester: *see* Yohimbine.

Et ester: $C_{22}H_{28}O_3N_2$. MW, 368. Cryst. M.p. 190°.

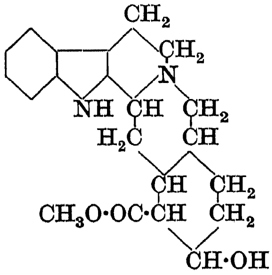
Propyl ester: $C_{23}H_{30}O_3N_2$. MW, 382. Cryst. + $1H_2O$. M.p. 137°.

Butyl ester: $C_{24}H_{32}O_3N_2$. MW, 396. Cryst. + $1H_2O$. M.p. 119–22°, anhyd. 127°.

Benzyl ester: $C_{27}H_{30}O_3N_2$. MW, 430. Amorp. powder from EtOH.Aq. M.p. 77–8°. *B,HCl*: cryst. powder from EtOH–Me_2CO. M.p. 253–4°.

Worrall, *J. Am. Chem. Soc.*, 1935, **57**, 900; 1933, **55**, 3715.
Field, *J. Chem. Soc.*, 1923, **123**, 3003.
Witkop, *Ann.*, 1943, **554**, 83, 127.

Yohimbine (*Quebrachine, hydroergotocin, aphrodine, corynine, methyl ester of yohimbic acid*)



$C_{21}H_{26}O_3N_2$ MW, 354

Chief alkaloid of *Corynanthe Johimbe*. Needles from EtOH.Aq. M.p. 241°. Sol. EtOH, $CHCl_3$, hot C_6H_6. Mod. sol. Et_2O. Spar. sol. H_2O. $[\alpha]_D^{20}$ + 107·9° in Py. Sublimes at 159°/0·01 mm.

B,HCl: plates. M.p. 302°. $[\alpha]_D^{22}$ + 103·3° in H_2O.

Nitrate: m.p. 276°.

Mono-acetyl: m.p. 150°. Sol. EtOH, Et_2O, $CHCl_3$ with green fluor.

O : N-*Diacetyl*: m.p. 183°.

Methiodide: plates from Me_2CO. M.p. 249–50°.

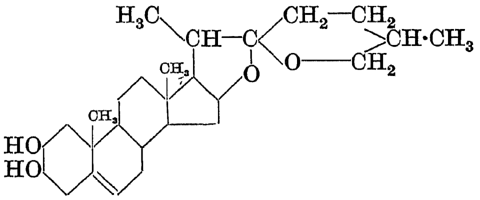
Robinson, *Annual Reviews on Biochemistry*, 1935, **4**, 504.
Warnat, *Ber.*, 1930, **63**, 2959.
Schomer, *Chem. Zentr.*, 1927, II, 2309.
Field, *J. Chem. Soc.*, 1923, **123**, 3003.
Witkop, *Ann.*, 1943, **554**, 83, 127.
Clemo, Swan, *J. Chem. Soc.*, 1946, 617; 1949, 487.

Isomeric forms of yohimbine are described in Heinemann, *Ber.*, 1934, 67, 18; Janot, Goutarel, *Bull. soc. chim.*, 1949, 659.

Yperite.

See 2 : 2′-Dichlorodiethyl sulphide.

Yuccagenin



$C_{27}H_{42}O_4$ MW, 430

Sapogenin isolated from various species of Yucca. Occurs as a tetraglycoside, yucconin. M.p. 252° (246°).

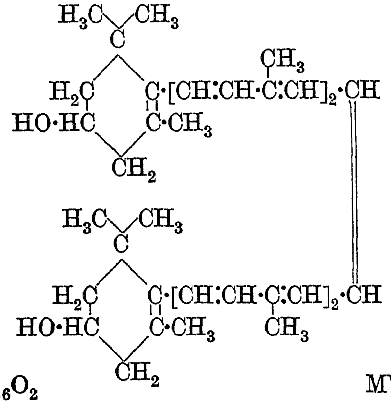
Acetyl deriv.: m.p. 178°.

Marker *et al.*, *J. Am. Chem. Soc.*, 1947, **69**, 2168, 2389, 2401.

Z

Zanthotoxin.
See Xanthotoxin.

Zeaxanthin



$C_{40}H_{56}O_2$ MW, 568

Constituent of lipochrome of *Fucus vesiculosus*, *Physalis*, egg yolk and spindle-tree maize. Yellow prisms from MeOH, cryst. + ½MeOH from MeOH–C_6H_6. M.p. 206·5° (215·5° corr.). Mod. sol. Et_2O, CS_2, C_6H_6, $CHCl_3$, CCl_4, Py, AcOEt. Spar. sol. pet. ether, MeOH. Absorption maxima at 515 mμ and 485 mμ in CS_2.

Mono-Me ether: $C_{41}H_{58}O_2$. MW, 582. Needles from MeOH. M.p. 153°.

Di-Me ether: $C_{42}H_{60}O_2$. MW, 596. Cryst. from MeOH. M.p. 176°.

Diacetyl: m.p. 154°.

Dipropionyl: cryst. from C_6H_6–MeOH. M.p. 142°.

Dibutyryl: cryst. from C_6H_6–MeOH. M.p. 132°.

Divaleryl: cryst. from C_6H_6–MeOH. M.p. 125°.

Dicaproyl: cryst. from C_6H_6–MeOH. M.p. 117–18°.

Dicaprylyl: cryst. from C_6H_6. M.p. 107°.

Dilauryl: m.p. 104°.

Monopalmityl: plates from C_6H_6–EtOH. M.p. 148°.

Dipalmityl: *see* Physalien.

Distearyl: m.p. 95°.

Karrer, Solmssen, *Helv. Chim. Acta*, 1935, **18**, 477.
Heilbron, Phipers, *Biochem. J.*, 1935, **29**, 1369.
Gillam, *ibid.*, 1831.
Kuhn, Grundmann, *Ber.*, 1934, **67**, 596.
Karrer, Wehrli, Helfenstein, *Helv. Chim. Acta*, 1930, **13**, 271.

Zebromal (*Ethyl ester of* trans-α : β-*dibromocinnamic acid*)

$C_6H_5{\cdot}CHBr{\cdot}CHBr{\cdot}CO{\cdot}OC_2H_5$

$C_{11}H_{12}O_2Br_2$ MW, 336

Plates. M.p. 75–6°. Sol. Et_2O, $CHCl_3$. Spar. sol. EtOH. Insol. H_2O. Boiling alc. KOH ⟶ phenylpropiolic acid. Used in treatment of epilepsy.

Merck, D.R.P., 271,434, (*Chem. Zentr.*, 1914, I, 1235).
Merck, *Pharm. Zentralhalle*, 1912, **53**, 591.
Anschütz, Kinnicutt, *Ber.*, 1878, **11**, 1220.

Zeorin

$C_{30}H_{52}O_2$ MW, 444

Triterpene from various lichens, e.g. *Anaptychia speciosa*, *A. hypoleuca*, *A. heterochroa*. Cryst. from Me_2CO. M.p. 223–7°. $[\alpha]_D$ + 54° in $CHCl_3$.

Mono-acetyl: m.p. 225–30°. $[\alpha]_D$ + 78° in $CHCl_3$.

Monobenzoyl: cryst. from C_6H_6–Me_2CO. M.p. 236–46°. $[\alpha]_D$ + 75° in $CHCl_3$.

Asahina, Yosioka, *Ber.*, 1940, **73**, 742.
Asahina, Akagi, *Ber.*, 1938, **71**, 980.
Barton, Bruun, *J. Chem. Soc.*, 1952, 1683.

Zierin (*Glucoside of cyanhydrin of* m-*hydroxybenzaldehyde*)

$C_{14}H_{17}O_7N$ MW, 311

Obtained from aerial parts of *Zieria lævigata*. Needles from AcOEt–$CHCl_3$. Softens at 153°, m.p. 156°. Sol. H_2O, EtOH, Me_2CO, MeCOEt. Very spar. sol. Et_2O, $CHCl_3$, pet. ether, C_6H_6. $[\alpha]_D^{20\cdot2}$ − 29·5°. $FeCl_3$ ⟶ faint blue col. Hyd. ⟶ glucose + HCN + *m*-hydroxybenzaldehyde.

Acetyl: cryst. from EtOH.Aq. M.p. 115–18°.

Finnemore, Cooper, *J. Proc. Roy. Soc. N.S. Wales*, 1936, **70**, 175, (*Chem. Zentr.*, 1937, I, 878).

Zierone

$C_{15}H_{22}O$ MW, 218

Constituent of volatile oil of *Zieria macrophylla*, Bonpland. Sesquiterpene ketone, isomeric with eremophilone. Viscous colourless oil. B.p. 147–9°/18 mm.

Semicarbazone: prisms from MeOH. M.p. 182°.

2 : 4-*Dinitrophenylhydrazone*: needles from EtOH. M.p. 95–7°.

Bradfield, Penfold, Simonsen, *J. Proc. Roy. Soc. N.S. Wales*, 1933, **67**, 200, (*Chem. Zentr.*, 1934, I, 1982).

Zinc dibutyl

$Zn(CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3)_2$

$C_8H_{18}Zn$ MW, 179·5

Liq. B.p. 193°, 81–2°/9 mm. D_4^{20} 1·034.

Noller, *Organic Syntheses*, 1932, XII, 86.
Hatch, Sutherland, Ross, *J. Org. Chem.*, 1949, **14**, 1130.

Zinc di-*sec*.-butyl

$Zn(CH(CH_3){\cdot}CH_2{\cdot}CH_3)_2$

$C_8H_{18}Zn$ MW, 179·5

Liq. B.p. 56°/4 mm.

Soroos, Morgana, *J. Am. Chem. Soc.*, 1944, **66**, 893.

Zinc diethyl (*Zinc ethyl*)

$Zn(C_2H_5)_2$

$C_4H_{10}Zn$ MW, 123·5

M.p. – 28°. B.p. 118°. D^{18} 1·1826. Spontaneously inflammable in air. $H_2O \longrightarrow C_2H_6 + Zn(OH)_2$. $NH_3 \longrightarrow Zn(NH_2)_2$. Hydroxy compounds $\longrightarrow$ ethane.

Noller, *Organic Syntheses*, 1932, XII, 86.

Zinc di-isobutyl

$Zn[CH_2{\cdot}CH(CH_3)_2]_2$

$C_8H_{18}Zn$ MW, 179·5

Liq. B.p. 185° (165–7°).

Ponzio, *Gazz. chim. ital.*, 1900, **30**, ii, 25.

Zinc di-isopropyl

$Zn[CH(CH_3)_2]_2$

$C_6H_{14}Zn$ MW, 151·5

Liq. B.p. 135°, 94–8°/40 mm. Fumes in air. Inflammable with difficulty in air and oxidised to $Zn(OC_3H_7)_2$.

Bohm, *Chem. Zentr.*, 1899, I, 1067.
Soroos, Morgana, *J. Am. Chem. Soc.*, 1944, **66**, 893.

Zinc dimethyl (*Zinc methyl*)

$Zn(CH_3)_2$

C_2H_6Zn MW, 95·5

M.p. – 42·5°. B.p. 46°. $D^{10·5}$ 1·386. Ignites spontaneously in air. $H_2O \longrightarrow CH_4 + Zn(OH)_2$.

Renshaw, Greenlaw, *J. Am. Chem. Soc.*, 1920, **42**, 1472.

Zinc diphenyl

$Zn(C_6H_5)_2$

$C_{12}H_{10}Zn$ MW, 219·5

Needles from C_6H_6. M.p. 105–6°. B.p. 280–5° slight decomp. Sol. Et_2O, C_6H_6. Spar. sol. pet. ether. Decomp. in dry air $\longrightarrow$ ZnO + diphenyl. $H_2O \longrightarrow Zn(OH)_2 + C_6H_6$. $CHCl_3 \longrightarrow$ triphenylmethane.

Hilpert, Grüttner, *Ber.*, 1913, **46**, 1680.

Zinc dipropyl (*Zinc propyl*)

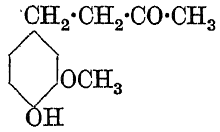
$Zn(CH_2{\cdot}CH_2{\cdot}CH_3)_2$

$C_6H_{14}Zn$ MW, 151·5

Liq. B.p. 146° (154°), 39–40°/9 mm. D_4^{20} 1·080. $H_2O \longrightarrow$ propane + $Zn(OH)_2$.

Noller, *Organic Syntheses*, 1932, XII, 86.
Hatch, Sutherland, Ross, *J. Org. Chem.*, 1949, **14**, 1130.

Zingerone (*4-Hydroxy-3-methoxybenzylacetone*)



$C_{11}H_{14}O_3$ MW, 294

Constituent of oil of ginger. Needles or plates with sweet odour from Et_2O–pet. ether. M.p. 40–1°. Spar. sol. H_2O, pet. ether. Sol. dil. alkalis. Slowly volatile in steam. Reduces $NH_3.AgNO_3$ in the warm. Alc. $FeCl_3 \longrightarrow$ green col.

Oxime: m.p. 87·5–88·5°.

Semicarbazone: needles. M.p. about 133°.

Phenylhydrazone: plates. M.p. about 143°.

Me ether: $C_{12}H_{16}O_3$. MW, 308. Needles from EtOH or MeOH. M.p. 55–6°. B.p. 186°/16 mm. Spar. sol. pet. ether. Insol. H_2O. *Oxime*: needles from Et_2O–pet. ether or MeOH.Aq. M.p. 93–4°.

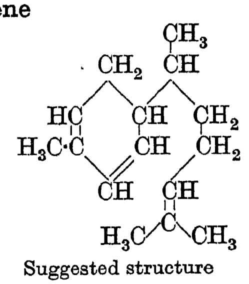
Et ether: $C_{13}H_{18}O_3$. MW, 322. Cryst. from EtOH.Aq. or pet. ether. M.p. 66°. Insol. H_2O.

Acetyl: cryst. M.p. 40–2°. B.p. 204–5°/14 mm.

Benzoyl: cryst. from EtOH. M.p. 126–7°.

Mannich, Merz, *Chem. Abstracts*, 1927, **21**, 1449.
Nomura, Can. P., 203,512 (*Chem. Abstracts*, 1920, **14**, 2936).
Lapworth, Pearson, Royle, *J. Chem. Soc.*, 1917, **111**, 777.

Zingiberene



Suggested structure

$C_{15}H_{24}$ MW, 204

Constituent of ginger oil. Natural oil is always contaminated with bisabolene. B.p. 134°/14 mm. D^{20} 0·8684. n_D^{20} 1·4956. $[\alpha]_D^{20}$ – 73·38°. Resinifies on standing. $HBr \longrightarrow$ dihydrobromide of isozingiberene. AcOH + $H_2SO_4 \longrightarrow$ isozingiberene. AcOH (+ Pt) $\longrightarrow$ hexahydrozingiberene. Heat with S $\longrightarrow$ cadalene.

Nitrosochloride: m.p. 93–4°.
Nitrosite: (i) M.p. 120–1°. (ii) M.p. 105°.
Nitrosate: m.p. 86–8°.

Ruzicka, van Veen, *Ann.*, 1929, **468**, 143.
Moudgill, *J. Indian Chem. Soc.*, 1928, **5**, 255.
Eschenmoser, Schinz, *Helv. Chim. Acta*, 1950, **33**, 171.

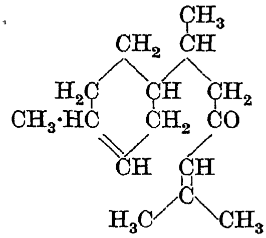
Zingiberol

$C_{15}H_{26}O$ MW, 222

Constituent of ginger oil. Liq. B.p. 154–7°/14·5 mm. HCl in AcOH ⟶ isozingiberene dihydrochloride.

Brooks, *J. Am. Chem. Soc.*, 1916, **38**, 431.

Zingiberone



Suggested structure

$C_{15}H_{24}O$ MW, 220

Liq. B.p. 156°/6 mm.

Mukherjee, *J. Indian Chem. Soc.*, 1948, **25**, 155.

Zoomaric Acid.
See Palmitoleic Acid.

Zygadenine

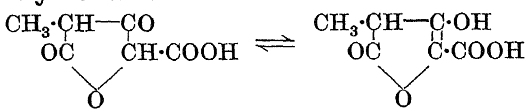
$C_{27}H_{43}O_7N$ MW, 493

Alkaloid from leaves of *Zygadenus intermedius* (Death camas). Cryst. from Et_2O, needles from C_6H_6, prisms + 2EtOH from EtOH. M.p. 201–4°. $[\alpha]_D^{24}$ − 45° in $CHCl_3$. Conc. H_2SO_4 ⟶ orange to red col. Physiological action resembles that of cevadine.

B,HCl: m.p. 231–4°.
B_2,H_2SO_4: m.p. 237–42°.
Hexa-acetyl deriv.: m.p. 265–70°.

Heyl, Hepner, Loy, *J. Am. Chem. Soc.*, 1913, **35**, 258.
Heyl, Herr, *J. Am. Chem. Soc.*, 1949, **71**, 1751.

Zymonic Acid



$C_6H_6O_5$ MW, 158

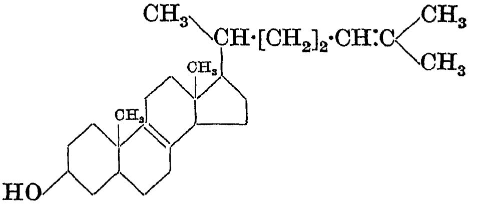
Product of incomplete fermentation of glucose by the yeasts *Trichosporon capitatum*, *Hansenula subpelliculosa* and *Kloeckera brevis*. Unstable. Resinifies and evolves CO_2 at room temp. $FeCl_3$ ⟶ red col.

Me ether: $C_7H_8O_5$. MW, 172. Cryst. + $1H_2O$ from damp di-*n*-butyl ether. M.p. 86–90°. Effervesces at 110–20°. *Me ester*: $C_8H_{10}O_5$. MW, 186. Oil. B.p. 118–23°/1 mm. n_D^{28} 1·4640. $[\alpha]_D^{27}$ + 1·2° in dimethylformamide. *Amide*: $C_7H_9O_4N$. MW, 171. Cryst. from Me_2CO–light petroleum. M.p. 209–10°. Unaffected by boiling acetic anhydride or by 2 : 4-dinitrophenylhydrazine. *Anilide*: $C_{13}H_{13}O_4N$. MW, 247. Cryst. from 80% EtOH. M.p. 127–127·5°.

Et ether: *amide*: $C_8H_{11}O_4N$. MW, 185. Cryst. from Me_2CO–light petroleum. M.p. 195·5–196·5°.

Stodola, Shotwell, Lockwood, *J. Am. Chem. Soc.*, 1952, **74**, 5415.

Zymosterol



$C_{27}H_{44}O$ MW, 384

The second most abundant sterol of yeast fat. M.p. 110°. $[\alpha]_D^{20}$ + 49° in $CHCl_3$.

Acetyl: m.p. 107–8°. $[\alpha]_D^{20}$ + 35° in $CHCl_3$.

Fieser, Fieser, *Natural Products Related to Phenanthrene*, 3rd. Ed., (Reinhold Publishing Corporation, New York), p. 289.
Heath-Brown, Heilbron, Jones, *J. Chem. Soc.*, 1940, 1482.

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